CONESTOGA-ROVERS & ASSOCIATES

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Quality Assurance Project Plan (QAPP)
Revision 01

South Dayton Dump and Landfill
Moraine, Ohio

Conestoga-Rovers & Associates
651 Colby Drive
Waterloo, Ontario N2V 1C2

November 2014 • 038443-14 • Report No. 29

Partners in Sustainability

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List of Acronyms and Short Forms

AA	Ambient Air
C	Celsius
CA	Corrective Action
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
COC	Chain of Custody
CRA	Conestoga-Rovers & Associates
CVAA	Cold Vapor Atomic Absorption
DO	Dissolved Oxygen
DOC	Dissolved Organic Carbon
DQIs	Data Quality Indicators
DQOs	Data Quality Objectives
EPA	Environmental Protection Agency
EDD	Electronic Data Deliverables
F	Fahrenheit
FSP	Field Sampling Plan
GC	Gas Chromatography
GC / ECD	Gas Chromatograph / Electron Capture Detector
GC / MC	Gas Chromatograph / Mass Spectroscopy
GPS	Global Positioning System
HDPE	High Density Polyethylene
IA	Indoor Air
IC	Ion Chromatography
ICP	Inductively Coupled Plasma
LCS	Laboratory Control Sample
LCS / LCSD	Laboratory Control Sample / Laboratory Control Sample Duplicate
LOQ	Limit of Quantitation
MDL	Method Detection Limit
mg / Kg	Milligram per Kilogram
mg / L	Milligram per Liter
MNA	Monitored Natural Attenuation
MS	Mass Spectroscopy
mS / cm	MilliSiemen per centimeter
MS / MSD	Matrix Spike / Matrix Spike Duplicate
mV	Millivolts
MW	Monitoring Well
NA	Not Applicable
NAPL	Non-aqueous Phase Liquid

List of Acronyms and Short Forms

NTU	Nephelometric Turbidity Units
ODH	Ohio Department of Health
PAL	Project Action Limit
PCBs	Polychlorinated Biphenyls
pg / g	Picogram per gram
pg / L	Picogram per Liter
PQOs	Project Quality Objectives
ppb v / v	Parts per Billion volume per volume
ppm	Parts per Million
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RPD	Relative Percent Difference
RL	Reporting Limit
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RT	Retention Time
Site	South Dayton Dump and Landfill Site
SOPs	Standard Operating Procedures
SU	Standard Units
SW-486	"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods",
EPA SW-486,	3rd Edition with Updates I through III, November 1986
SVOC	Semi-Volatile Organic Compounds
TAL	Target Analyte Group
TBD	To Be Determined
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
TOC	Total Organic Carbon
TA	TestAmerica, Inc.
TA – NC	TestAmerica North Canton, Ohio
TA – KX	TestAmerica Knoxville, Tennessee
TQL	Target Quantitation Limit
µg / Kg	Microgram per Kilogram
µg / L	Microgram per Liter
USEPA	United States Environmental Protection Agency

List of Acronyms and Short Forms

VAS	Vertical Aquifer Sampling
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compounds

QAPP Worksheet #1 - Title and Approval Page

Site Name/Project Name:	South Dayton Dump and Landfill
Site Location:	South Dayton Dump and Landfill 2139 Dryden Road Moraine, Ohio 45439
Document Title:	Quality Assurance Project Plan
Lead Organization & Federal Regulatory Agency	USEPA Region 5
Preparer's Name, Organization, and Contact Information:	Steve Quigley Conestoga-Rovers & Associates 651 Colby Drive, Waterloo, Ontario, Canada, N2V 1C2 (519) 884-0510 squigley@craworld.com
Preparation Date:	
Investigative Organization's Project Manager For Investigative Activities:	Signature:_____ Date:_____ Steve Quigley, CRA
Investigative Organization's Quality Assurance Officer:	Signature:_____ Date:_____ Angela Bown, CRA
Lead Organization's Program Manager:	Signature:_____ Date:_____ Leslie Patterson, USEPA Region 5
TestAmerica – North Canton Laboratory Project Manager:	Signature:_____ Date:_____ Denise Heckler, TA-NC
TestAmerica – North Canton Laboratory Quality Assurance Officer:	Signature:_____ Date:_____ Dorothy Leeson, TA-NC
TestAmerica – Knoxville Laboratory Project Manager:	Signature:_____ Date :_____ Jamie McKinney, TA-KX

TestAmerica – Knoxville Laboratory Quality Assurance Officer:	Signature:_____ Kevin McGee, TA-KX	Date :_____
USEPA Quality Assurance Reviewer:	Signature:_____ Warren Layne, USEPA Region 5	Date:_____
Ohio EPA Project Manager:	Signature:_____ Madelyn Smith, Ohio EPA	Date:_____

QAPP Worksheet #2 - Identifying Information

Site Name/Project Name:	South Dayton Dump and Landfill
Site Location:	South Dayton Dump and Landfill 2139 Dryden Road Moraine, Ohio 45439
Site Number/Code:	EPA ID# OHD980611388
Operable Unit:	Operable Units 1 and 2 (OU1 and OU2)
Contractor's Name:	Not Applicable
Contractor's Number:	Not Applicable
Contract Title:	Not Applicable
Work Assignment Number:	Not Applicable
Identify guidance used to prepare QAPP:	Uniform Federal Policy for Quality Assurance Project Plans (Intergovernmental Data Quality Task Force, 2005a)
Identify regulatory program:	Comprehensive Environmental Response, Compensation and Liability Act (CERCLA)
Identify approval entity:	USEPA Remedial Program, Region 5
Indicate whether the QAPP is a generic or a project-specific QAPP:	Site-specific Generic QAPP for the South Dayton Dump and Landfill Site
List dates of scoping sessions that were held:	Original scoping sessions were held between 2004 and 2006. Additional meetings that included discussion of project scope have been held since that time as needed.
List dates and titles of QAPP documents written for previous site work, if applicable:	QAPP (CRA, 2008) QAPP (CRA, 2011)
List data users:	Risk Assessors, Chemists, Statisticians, Geologists, Hydro-geologists
Lead Organization's Program Manager:	Leslie Patterson, USEPA

REQUIRED QAPP ELEMENT(S) AND CORRESPONDING QAPP SECTION(S) (USEPA, 2005a)	*REQUIRED INFORMATION*	*CROSSWALK TO RELATED INFORMATION AND DOCUMENTS*
Project Management and Objectives		
2.1 Title and Approval Page	• Title and Approval Page	Worksheet #1, Title and Approval Page
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information	• Table of Contents • QAPP Identifying Information	The Table of Contents is provided following the QAPP cover page. Worksheet #2, Identifying Information
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	• Distribution List • Project Personnel Sign-Off Sheet	Worksheet #3, Distribution List; and Worksheet #4, Project Personnel Sign-Off Sheet
2.4 Project Organization 2.4.1 Project Organization Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	• Project Organizational Chart • Communication Pathways • Personnel Responsibilities and Qualifications Table • Special Personnel Training Requirements Table	Worksheet #5, Project Organization Charts; Worksheet #6, Communication Pathways; Worksheet #7, Personnel Responsibilities and Qualifications; Worksheet #8, Special Personnel Training Requirements
2.5 Project Planning/Problem Definition 2.5.1 Project Planning (Scoping) 2.5.2 Problem Definition, Site History, and Background	• Project Planning Session • Documentation (including Data Needs tables) • Problem Definition, Site History, and Background • Site Maps (historical and current)	Worksheet #10, Conceptual Site Model *Site History and more details concerning the project DQOs can be found in the previous reports:* • *Remedial Investigation Report: Operable Unit 1 (CRA, 2010)* • *Streamlined Feasibility Study (CRA, 2010)* • *Vapor Intrusion Investigation Summary Report (CRA, 2012)* • *VI Mitigation Work Plan (CRA, 2013)* • *Operable Unit Two (OU2) Remedial Investigation/Feasibility Study (RI/FS) Work Plan (CRA, 2014).*
2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process	• Site-Specific Project Quality Objectives (PQOs) • Measurement Performance Criteria	Worksheets #11-1 through #11-6, Project/Data Quality Objectives; Worksheet #12-1 through #12-19, Measurement Performance Criteria

REQUIRED QAPP ELEMENT(S) AND CORRESPONDING QAPP SECTION(S) (USEPA, 2005a)	*REQUIRED INFORMATION*	*CROSSWALK TO RELATED INFORMATION AND DOCUMENTS*
2.6.2 Measurement Performance Criteria	Table	
2.7 Secondary Data Evaluation	• Sources of Secondary Data and Information • Secondary Data Criteria and Limitations Table	Worksheet #13, Secondary Data Criteria and Limitations
2.8 Project Overview and Schedule 2.8.1 Project Overview 2.8.2 Project Schedule	• Summary of Project Tasks • Reference Limits and Evaluation Table • Project Schedule/Timeline Table	Worksheet #14, Summary of Project Tasks; Worksheet #15-1 through #15-6, Reference Limits and Evaluation; Worksheet #16, Project Schedule/Timeline
Measurement/ Data Acquisition		
3.1 Sampling Tools 3.1.1 Sampling Process Design and Rationale 3.1.2 Sampling Procedures and Requirements 3.1.2.1 Sampling, Collection Procedures 3.1.2.2 Sample Containers, Volume, and Preservation 3.1.2.3 Equipment/Sample Container Cleaning and Decontamination Procedures 3.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection procedures 3.1.2.5 Supply and Inspection and Acceptance Procedures 3.1.2.6 Field Documentation Procedures	• Sampling Design and Rationale • Sample Location Map • Sampling Locations and Methods/SOP Requirements Table • Analytical Methods/SOP Requirements Table • Field Quality Control Sample Summary Table • Sampling SOPs • Project Sampling SOP References Table • Field Equipment Calibration, Maintenance, Testing, and Inspection Table	Worksheet #17, Sampling Design and Rationale; Worksheet #18, Sampling Locations and Methods/SOP Requirements; Worksheet #19, Analytical SOP Requirements (sample containers, preservation, and holding times); Worksheet #20, Field Quality Control Sample Summary Worksheet #21, Project Sampling SOP Reference; Worksheet #22, Field Equipment Calibration, Maintenance, Testing, and Inspection *The laboratory SOPs can be found as listed below:* *Laboratory Sample Analysis SOPs in Appendix D* *Laboratory Sample Preparation SOPs in Appendix E* *Laboratory Support SOPs in Appendix F.* *More details concerning the sampling design and rationale and the field sampling procedures can be found in the previous reports:* • *Field Sampling Plan (CRA, 2013)* • *VI Mitigation Work Plan (CRA, 2013)* • *Operable Unit Two (OU2) Remedial*

REQUIRED QAPP ELEMENT(S) AND CORRESPONDING QAPP SECTION(S) (USEPA, 2005a)	***REQUIRED INFORMATION***	***CROSSWALK TO RELATED INFORMATION AND DOCUMENTS***
		Investigation/Feasibility Study (RI/FS) Work Plan (CRA, 2014).
3.2 Analytical Tools 3.2.1 Analytical SOPs 3.2.2 Analytical Instruments 3.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures 3.2.4 Analytical Supply Inspection and Acceptance Procedures	• Analytical SOPS • Analytical SOP Reference Table • Analytical Instrument Calibration Table • Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	Worksheets #23-1 through #23-3, Analytical SOP References; Worksheet #24, Analytical Instrument Calibration; Worksheet #25, Analytical Instrument and Equipment Maintenance, Testing, and Inspection *The laboratory SOPs can be found as listed below:* *Laboratory Sample Analysis SOPs in Appendix D* *Laboratory Sample Preparation SOPs in Appendix E* *Laboratory Support SOPs in Appendix F.*
3.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures 3.3.1 Sample Collection Documentation 3.3.2 Sample Handling and Tracking System 3.3.3 Sample Custody	• Sample Collection Documentation Handling, Tracking, and Custody SOPs • Sample Container Identification • Sample Handling Flow Diagram • Example Chain-of-Custody Record and Seal	Worksheet #26, Sample Handling System; Worksheet #27, Sample Custody Requirements More details concerning the field sampling procedures can be found in the previous reports: • *Field Sampling Plan (CRA, 2013)* • *VI Mitigation Work Plan (CRA, 2013)* • *Operable Unit Two (OU2) Remedial Investigation/Feasibility Study (RI/FS) Work Plan (CRA, 2014).* Example Chain-of-Custody (COC) forms can be found in Appendix C
3.4 Quality Control Samples 3.4.1 Sampling Quality Control Samples 3.4.2 Analytical Quality Control Samples	• QC Samples Table • Screening/Confirmatory Analysis Decision Tree	Worksheets #28-1 through #28-18, Present QC sample information for project analysis
3.5 Data Management Tools 3.5.1 Project Documentation and Records 3.5.2 Data Package Deliverables 3.5.3 Data Reporting Formats 3.5.4 Data Handling and Management	• Project Documents and Records Table • Analytical Services Table • Data Management SOPs	Worksheet #29, Project Documents and Records, Worksheet #30, Analytical Services

REQUIRED QAPP ELEMENT(S) AND CORRESPONDING QAPP SECTION(S) (USEPA, 2005a)	*REQUIRED INFORMATION*	*CROSSWALK TO RELATED INFORMATION AND DOCUMENTS*
3.5.5 Data Tracking and Control		
4.1 Assessments and Response Actions 4.1.1 Planned Assessments 4.1.2 Assessment Findings and Corrective Action Response	• Assessments and Response Actions • Planned Project Assessments Table Audit Checklists • Assessment Findings and Corrective Action Responses Table	Worksheet #31, Planned Project Assessments, Worksheet #32, Assessment Findings and Corrective Action Responses *The laboratory Quality Assurance Manual and Laboratory Policies and Guidelines documents can be found in Appendix F*
4.2 QA Management Reports	• QA Management Reports Table	Worksheet #33, QA Management Reports
4.3 Final Project Report		
Data Review		
5.1 Overview		
5.2 Data Review Steps 5.2.1 Step I: Validation 5.2.2 Step II: Validation 5.2.2.1 Step IIa Validation Activities 5.2.2.2 Step IIb Validation Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities	• Inputs to Data review process • Validation (Steps IIa and IIb) Process Table • Validation Summary Table • Usability Assessment	Worksheet #34, Data Verification and Validation Inputs; Worksheet #35, Data Verification and Validations Procedures; Worksheet #36,Validation Summary; Worksheet #37, Usability Assessment
5.3 Streamlining Data Review 5.3.1 Data Review Steps To Be Streamlined 5.3.2 Criteria for Streamlining Data Review 5.3.3 Amounts and Types of Data Appropriate for Streamlining	• None	N/A

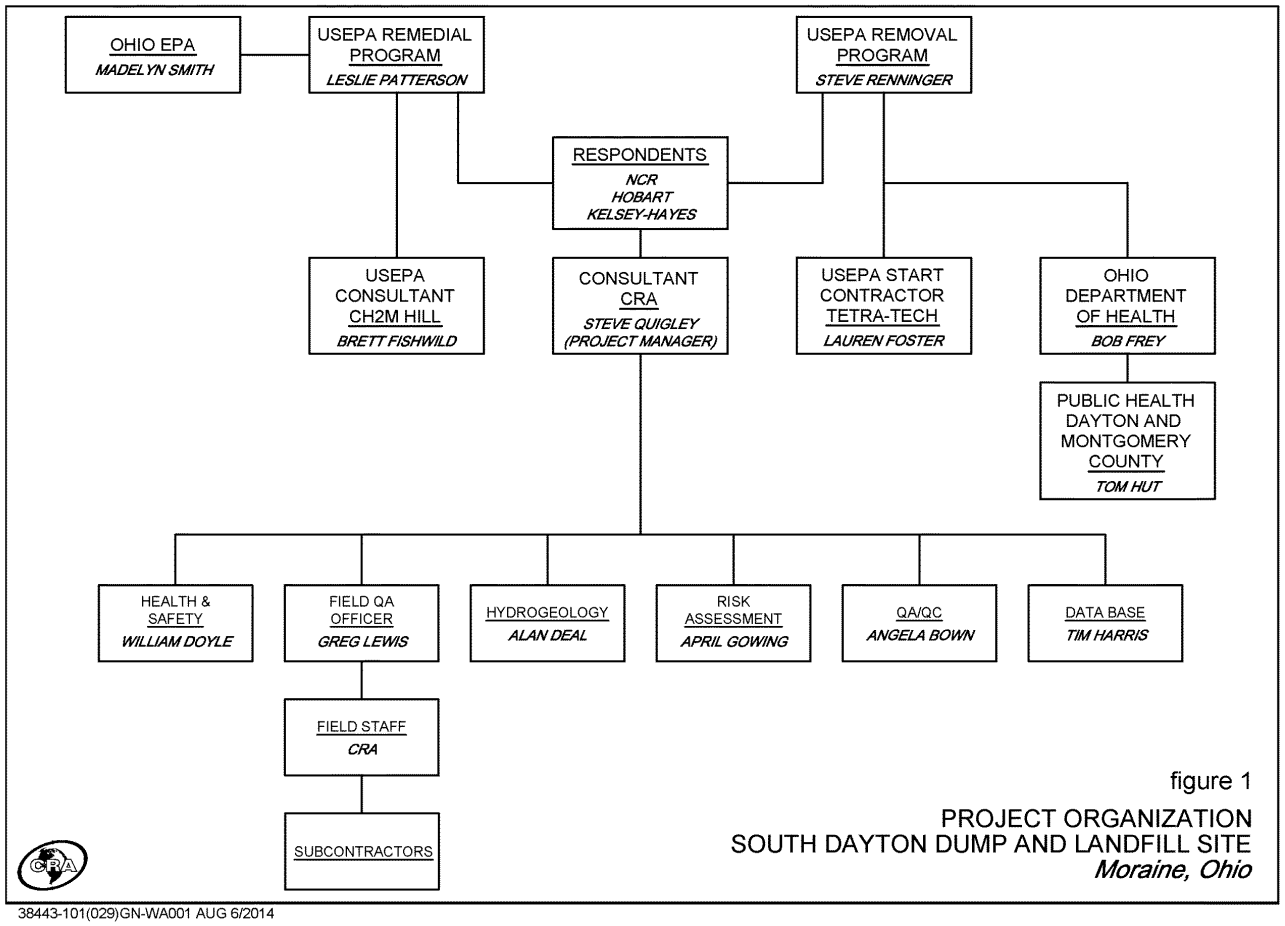
QAPP Worksheet #3 - Distribution List

QAPP RECIPIENTS	*TITLE*	*ORGANIZATION*	*TELEPHONE NUMBER*	*E-MAIL ADDRESS*	*DOCUMENT CONTROL NUMBER*
Leslie Patterson	USEPA Remedial Program Project Manager	USEPA, Region 5	312 – 886 – 4904	patterson.leslie@epa.gov	
Warren Layne	USEPA Region 5 QAPP Reviewer	USEPA, Region 5	312 – 886 – 7336	layne.warren@epa.gov	
Madelyn Smith	Ohio EPA Project Manager	Ohio EPA	937 – 285 – 6456	Madelyn.smith@epa.ohio.gov	
Jim Campbell		Engineering Management, Inc.	412 – 244 – 0917	jrc@e-emi.com	
Ken Brown		Illinois Tool Works Inc. (ITW)	847 – 657 – 4843	kbrown@itw.com	
Wendell Barner		Kelsey-Hayes Co.	412 – 339 – 4775	wendell.barner@gmail.com	
Bryan Heath		NCR	678 – 808 – 6061	bryan.heath@ncr.com	
Steve Quigley	Project Manager	CRA	519 – 884 – 0510	squigley@craworld.com	
Angela Bown	QA Officer	CRA	513 – 942 – 4750	abown@craworld.com	
Denise Heckler	Laboratory Project Manager	TA – NC	330 – 966 – 9477	denise.heckler@testamericainc.com	
Jamie McKinney	Laboratory Project Manager	TA - KX	865 – 291 – 3051	jamie.mckinney@testamericainc.com	

QAPP Worksheet #4 & 7 - Personnel Qualifications and Sign-off Sheet

ORGANIZATION: United State Environmental Protection Agency Region 5			
Name	**Project Title/Role**	**Education/Experience**	**Signature/Date**
Leslie Patterson	U.S.EPA Remedial Program Manager		
Warren Layne	USEPA Region 5 QAPP Reviewer		
ORGANIZATION: Ohio Environmental Protection Agency			
Name	**Project Title/Role**	**Education/Experience**	**Signature/Date**
Madelyn Smith	Ohio EPA Project Manager		
ORGANIZATION: Conestoga-Rovers & Associates			
Name	**Project Title/Role**	**Education/Experience**	**Signature/Date**
Steve Quigley	Project Manager	B.Sc. Environmental Engineering, University of Guelph, 1996 Over 14 years experience	
Angela Bown	QA Officer	B.S. Environmental Management, University of Findlay, 1999 Over 20 years experience	
ORGANIZATION: TestAmerica North Canton			
Name	**Project Title/Role**	**Education/Experience**	**Signature/Date**
Denise Heckler	Laboratory Project Manager	B.S. Chemistry, Youngstown State University, 1988 Over 20 years experience	
Dorothy Leeson	Laboratory QA Officer	B.S. Chemistry, Ohio University, 1984 Over 20 years experience	
ORGANIZATION: TestAmerica Knoxville			
Name	**Project Title/Role**	**Education/Experience**	**Signature/Date**
Jamie McKinney	Laboratory Project Manager		
Kevin McGee	Laboratory QA Officer		

QAPP Worksheet #5 - Project Organization Charts



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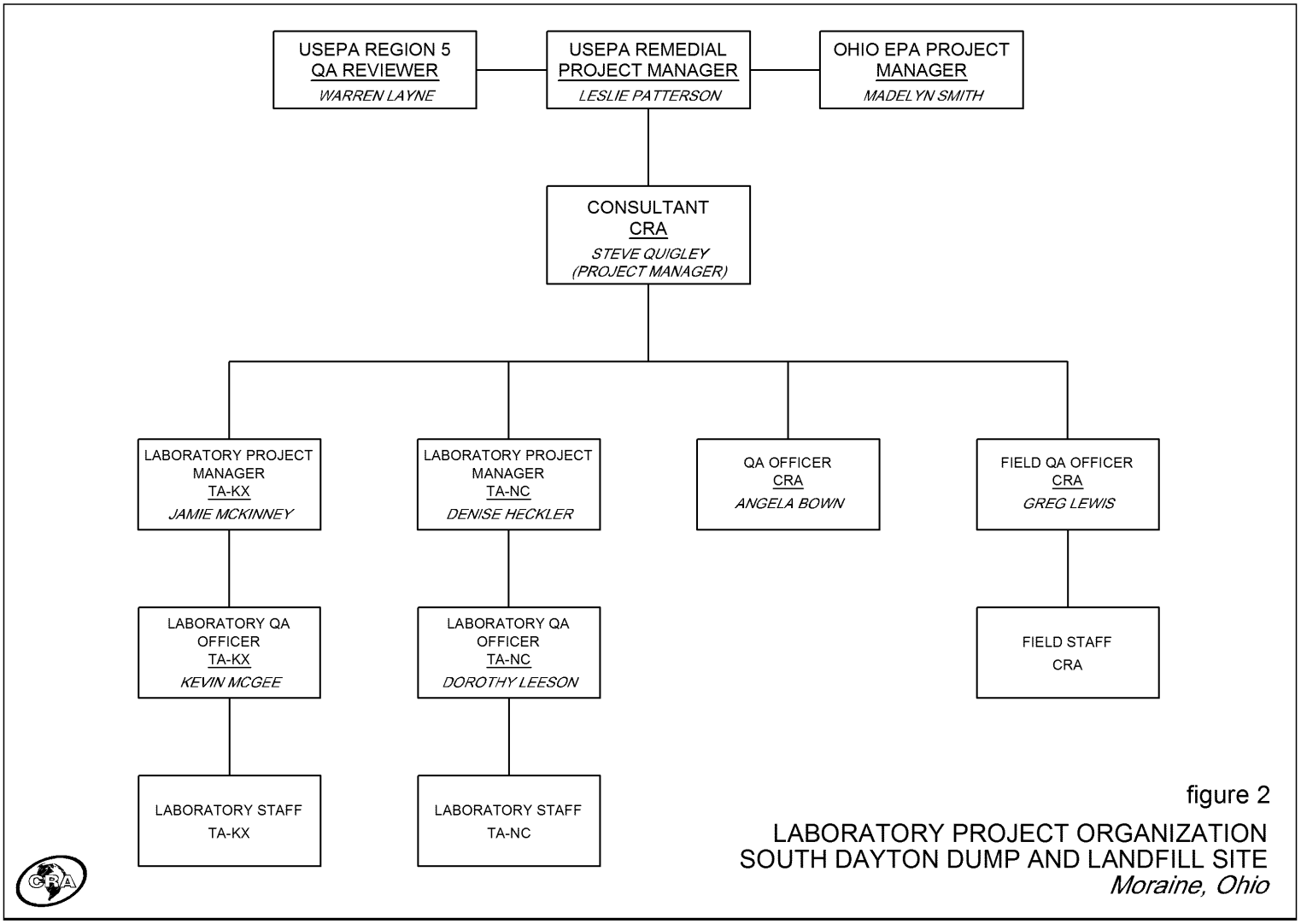

figure 2

LABORATORY PROJECT ORGANIZATION
SOUTH DAYTON DUMP AND LANDFILL SITE
Moraine, Ohio

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QAPP Worksheet #6 - Communication Pathways

COMMUNICATION DRIVERS	*RESPONSIBLE ENTITY*	*NAME*	*TELEPHONE NUMBER*	*PROCEDURE (timing, pathways etc.)*
Point of Contact with USEPA RPM	Project Manager (PM) CRA	Steve Quigley	519-884-0510	CRA's PM will be the liaison to the USEPA Regional Project Manager (RPM) for all activities at the South Dayton Dump and Landfill Site.
Stop work due to safety issues	Project Manager or Field Technicians, CRA USEPA oversight personnel	Steve Quigley Greg Lewis Jeremy Teepen Jason Close Nate Ziegler *USEPA oversight personnel – CH2M Hill*	519-884-0510 513-200-8902 513-309-5524 513-478-5021 513-476-5418 *[Phone number]*	Any project/field personnel has the authority to stop work should he/she identify unsafe conditions. Field Technicians will notify the PM regarding any implemented stop work authority as soon as possible. The PM will notify the Health and Safety Manager, Respondents, and USEPA, as appropriate.
Initiate and/or notify of changes or delays to field work	Project Manager or Field Technicians, CRA USEPA oversight personnel	Steve Quigley Greg Lewis Jeremy Teepen Jason Close Nate Ziegler *USEPA oversight personnel – CH2M Hill*	519-884-0510 513-200-8902 513-309-5524 513-478-5021 513-476-5418 *[Phone number]*	If field sampling will be delayed or changes to the field sampling procedures are requested, then the CRA field technicians will notify the CRA PM, who will then notify USEPA and Respondents, or request approval from USEPA in the case of proposed changes to sampling procedures.
Issue real-time approval of field work modifications	USEPA oversight personnel	*USEPA oversight personnel – CH2M Hill*	*[Phone number]*	As USEPA's contractor, CH2M Hill oversight personnel have the authorization to approve of field work modifications on behalf of USEPA as they arise. CRA field technicians will communicate the field work modifications to CRA's PM who will have the authority to approve field work modifications provided USEPA also approves of the modifications.
QAPP changes	PM, CRA	Steve Quigley	519-884-0510	Any requested changes to the QAPP will be sent by CRA's PM to the USEPA RPM for review and approval.

COMMUNICATION DRIVERS	*RESPONSIBLE ENTITY*	*NAME*	*TELEPHONE NUMBER*	*PROCEDURE (timing, pathways etc.)*
Sample receipt variances	Laboratory Manager, TestAmerica Inc.	Denise Heckler Jamie McKinney	330-966-9477 865-291-3051	The contracted laboratory PM will communicate any sample receipt issues within 2 business days of receipt, to CRA's QA/QC Officer. CRA's QA/QC Officer will determine the need for corrective action for field and analytical issues, in conjunction with the CRA PM or the Laboratory Project Manager, as appropriate. Corrective field actions will be communicated to the field technicians by the CRA PM.
Report issues related to analytical data quality, including ability to meet reporting limits	QA/QC Officer, CRA	Angela Bown	513-942-4750	CRA's QA/QC Officer will identify any issues related to analytical data quality in data validation memos that are submitted to CRA's PM CRA's QA/QC Officer will provide CRA's database project manager with the qualifications required for the analytical data.
Data validation & verification issues (e.g. incomplete records, non-compliance with procedures)	QA/QC Officer, CRA	Angela Bown	513-942-4750	CRA's QA/QC Officer will provide data validation memos to CRA's PM. CRA's QA/QC Officer will provide CRA's database project manager with the qualifications required for the analytical data.
Provide preliminary and validated data to managers and users	PM, CRA	Steve Quigley	519-884-0510	CRA's PM will provide preliminary data to USEPA, Respondents, and data users within 3 days of sample receipt. CRA's PM will provide validated data to USEPA, Respondents, and data users once approved by CRA's QA/QC Officer.
Provide validated data to the public	RPM, USEPA	Leslie Patterson	312-886-4904	USEPA's RPM will provide validated data to the public via the USEPA South Dayton Dump and Landfill Site website.
Provide assistance with Site Specific Health and Safety	Regional Safety & Health Manager (RSHM), CRA	William Doyle, CRA	734-357-5517	CRA's RSHM will provide assistance to CRA's PM and Field QA Officer. CRA's RSHM will review the safety records of any prospective subcontractor prior to notice of award.

COMMUNICATION DRIVERS	*RESPONSIBLE ENTITY*	*NAME*	*TELEPHONE NUMBER*	*PROCEDURE (timing, pathways etc.)*
Data Management	Project Manager, CRA Field Technicians, CRA Database Manager, CRA	Steve Quigley Greg Lewis Jeremy Teepen Jason Close Nate Ziegler Tim Harris	519-884-0510 513-942-4750 513-942-4750 513-942-4750 513-942-4750 519-884-0510	The CRA Field Technicians will provide field notes and sample log sheets to the CRA PM. The CRA PM will consolidate the sample location, sample ID and analysis information into a field sampling key, which will be provided to the CRA database manager. The CRA PM will provide the CRA database manager with information on the types of tables required.
Establish and maintenance of project database	Database Manager, CRA	Tim Harris	519-884-0510	CRA's database manager will provide requested analytical data to CRA project personnel

QAPP Worksheet #8 - Special Personnel Training Requirements

PROJECT FUNCTION	*SPECIALIZED TRAINING-TITLE OR DESCRIPTION OF COURSE*	*TRAINING PROVIDER*	*TRAINING DATE*	*PERSONNEL/GROUPS RECEIVING TRAINING*	*PERSONNEL TITLES/ORGANIZATIONAL AFFILIATION*	*LOCATION OF TRAINING RECORDS/CERTIFICATES*
Field Activities	40-hr HAZWOPER and Annual 8-hr refresher	Certified training professionals	Various	All CRA Field and subcontractor personnel that will be onsite	CRA personnel, subcontractors	CRA Employee Training Database
Sample Collection	Trained in USEPA CERCLA and CRA sampling methods and field testing procedures	CRA Class and On-Site field training	Various	All field personnel that perform sample collection	All field personnel that perform sample collection	CRA Employee Training Database
Sample Analysis	Trained in applicable analytical methods	Laboratory on-Site and vendor training	Various	Laboratory personnel	Laboratory personnel	Laboratory

TABLE 10.3
ECOLOGICAL CONCEPTUAL SITE MODEL
OPERABLE UNIT 1 AND 2 PARCELS
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

PRIMARY SOURCE	release mechanism	SECONDARY SOURCE	release mechanism	TERTIARY SOURCE	release mechanism	EXPOSURE ROUTE	RECEPTOR CHARACTERIZATION											
							POTENTIALLY EXPOSED RECEPTORS (ECOLOGICAL / HUMAN HEALTH - BASELINE CONDITIONS)											
							OU1 Parcels		OU2 Parcels (excluding Quarry Pond)		OU2 Quarry Pond			Off-site properties		Great Miami River / floodplain		
							Terrestrial Biota	Aquatic Biota	Terrestrial Biota	Aquatic Biota	Terrestrial Biota	Aquatic Biota	Humans that consume fish	Terrestrial Biota	Aquatic Biota	Terrestrial Biota	Aquatic Biota	Humans that consume fish
SURFACE LANDFILL CONTENTS (within OU1 Parcels)	direct contact					INGESTION	X	X	na	na	na	na	na	na	na	na	na	na
	plant uptake	VEGETATION	direct contact			INGESTION	X	X	na	na	na	na	na	na	na	na	na	na
	stormwater runoff	SURFACE WATER AND SEDIMENT	direct contact			INGESTION	X	X	(a)	(a)	(a)	(a)	--	X	X	X	X	--
			direct contact	AQUATIC ORGANISMS		INGESTION	X	X	(a)	(a)	(a)	(a)	--	X	X	X	X	X
SURFACE LANDFILL CONTENTS (within OU2 Parcels)	direct contact					INGESTION	na	na	X	--	--	X	--	na	na	na	na	na
	plant uptake	VEGETATION	direct contact			INGESTION	na	na	X	--	--	X	--	na	na	na	na	na
	stormwater runoff	SURFACE WATER AND SEDIMENT	direct contact			INGESTION	(a)	(a)	X	X	--	--	--	X	X	X	X	--
			direct contact	AQUATIC ORGANISMS		INGESTION	(a)	(a)	X	X	--	--	--	X	X	X	X	X
	stormwater runoff and infiltration	QUARRY POND	direct contact			INGESTION	na	na	na	na	X	X	--	na	na	na	na	na
			direct contact	AQUATIC ORGANISMS		INGESTION	na	na	na	na	X	X	X	na	na	na	na	na

LEGEND

--	incomplete exposure pathway e.g., due to absence of exposure route and/or receptor
na	not applicable due to spatial separation
(a)	potential cross-boundary effects between OU1 Parcels and OU2 Parcels will be considered in the OU2 RI/FS
X	potentially complete exposure pathway to be evaluated/addressed as part of OU1
X	potentially complete exposure pathway to be evaluated for OU2

QAPP Worksheet #11 - Project /Data Quality Objectives

11-1 - Process Soil and Fill

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Residential and Industrial Soil Criteria***	***Comparison to Background Reference Conditions***	***Additional sampling (if necessary) to develop risk assessment exposure estimates***
Step 1. State the Problem			
i) Problem Description	Insufficient soil quality data exist for OU2 in order to determine: - The nature and extent of contaminated soil and fill. - The nature and lateral and vertical extent of the contaminated soil and fill material.	- Insufficient soil quality data exist for OU2 in order to determine whether contaminant concentrations are from historic site activities or are due to elevated background concentrations (either naturally occurring or anthropogenic regional contamination).	If soil or fill containing site-related contaminants of concern (COCs) at concentrations greater than screening values and background reference conditions is found in Phases 1A and 1B for Southern Parcels, there may still be insufficient data to establish the presence or absence of direct contact, ingestion, and inhalation risks to receptors via soil and/or fill exposure pathways.
ii) Planning team	See note at bottom		
iii) Conceptual model	Fill was placed in a portion of the Southern Parcels. The fill includes but may not be limited to CDD. The fill may contain contaminants. OU2 soil may have site-related contaminants from wind-blown deposition, run-off, groundwater leaching, and/or re-depositing of contamination (i.e., regrading). - Contaminants in soil may pose a risk to human receptors via the direct contact, inhalation and ingestion pathways. Cover material at the Site is limited or non-existent, which could lead to erosional run-off of contaminants towards the Quarry Pond, which may pose a risk to human receptors and ecological receptors (e.g. wildlife, aquatic organisms) - Infiltrating precipitation can cause contaminants in soil and fill to migrate downwards, ultimately impacting groundwater. - Groundwater migrating from OU1 could deposit contaminants in the soil and/or fill of OU2.		
iv) General intended use for data	The soil and fill data collected will be compared to USEPA Residential and Industrial Soil Regional Screening Levels (RSLs) to identify direct contact/ingestion/inhalation human health risks, and compared to USEPA RCRA Ecological Screening Levels (ESLs) (USEPA, 2003) to identify ecological risks associated with soil and fill in OU2. The data collected will ultimately be used in the Remedial Investigation Report and Baseline Risk Assessment for OU2.	The data collected from sampling locations in the Southern Parcels will be compared to background conditions, to determine if there are measurable levels of Site-related contaminants. The data collected will ultimately be used in the Baseline Risk Assessment for OU2.	The collected data will be used to generate exposure estimates for an assessment of direct contact/ingestion/inhalation human health risks and risks to ecological receptors. The data collected will ultimately be used in the Baseline Human Health Risk Assessment and Ecological Risk Assessment for OU2.

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Residential and Industrial Soil Criteria***	***Comparison to Background Reference Conditions***	***Additional sampling (if necessary) to develop risk assessment exposure estimates***
Step 1. State the Problem			
v) Resources, constraints, deadlines	Sufficient resources will be committed to sample soil on the Southern Parcels under the OU2 RI/FS work plan. Sampling may be postponed due to flooding.		
Step 2. Goals of the Study			
i) Primary study question	Do soil and fill samples from the Southern Parcels contain contaminants at concentrations greater than Industrial or Residential Soil RSLs, or USEPA RCRA ESLs[1]?	Are contaminant concentrations due to Site activities or locally occurring background concentrations?	Does soil or fill in OU2 contain Site-related contaminants that pose unacceptable human health risks or unacceptable risks to ecological receptors?
ii) Alternate outcomes or actions	- If sampling demonstrates that contaminant concentrations in soil and fill are less than RSLs, no further sampling or remedial action is planned. - If sampling demonstrates that contaminant concentrations in soils or fill are greater than screening levels/criteria, further evaluation is needed to determine if the contamination is Site-related, and is a risk to human health and the environment, and/or remedial measures.	- If sampling demonstrates that contaminant concentrations in OU2 are not greater than those found in background reference soils, no further sampling is planned. - If statistical analysis indicates that additional sampling is required to obtain the necessary precision and accuracy, additional background samples will be collected.	- If sampling demonstrates that human health and ecological risks from all combined exposure pathways are acceptable, no further action is required. - If sampling demonstrates unacceptable human health or ecological risks, further evaluation, risk management and/or remediation would be required.
iii) Type of problem (decision or estimation)[1]	Decision (Action Level)	Decision (Action Level)	Estimation
iv.a) Decision statement	Determine whether any Site-related contaminant concentrations in soil and fill are greater than USEPA Industrial/Residential Soil RSLs, or USEPA RCRA ESLs in OU2.	Determine whether any measurable levels of Site-related contaminants, relative to background reference conditions, occur in soil and fill in OU2.	Determine where contaminant concentrations require further consideration or response action, and where no further investigation is necessary.

[1] Ecological Screening Levels (ESLs) will be presented and defined in the Screening Level Environmental Risk Assessment (SLERA) work plan

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Residential and Industrial Soil Criteria***	***Comparison to Background Reference Conditions***	***Additional sampling (if necessary) to develop risk assessment exposure estimates***
Step 1. State the Problem			
iv.b) Estimation statement & assumptions	--	--	The parameter of interest is the mean (for estimating direct contact/ingestion/inhalation risks) of soil/fill contaminant concentrations within identified exposure areas in OU2. The exposure areas are defined in Section 5.2 of the OU2 RI/FS Work Plan. The statistical measure of interest is the 95% UCL of the mean for each exposure unit. The size and location of each exposure unit has been identified based on property ownership boundaries and current and reasonably foreseeable activities and land uses.

[1] Ecological Screening Levels (ESLs) will be presented and defined in the Screening Level Environmental Risk Assessment (SLERA) work plan

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Residential and Industrial Soil Criteria***	***Comparison to Background Reference Conditions***	***Additional sampling (if necessary) to develop risk assessment exposure estimates***
Step 3. Identify Information Inputs			
i) Information types needed	- Identification and chemical analysis of soil and fill in OU2. - Contaminant concentrations in soil and fill in OU2. - Background soil contaminant concentrations. - Soil samples will be collected on a random basis (random oriented grid) from each exposure area; however, CRA will ensure that samples are collected from areas where geophysical anomalies have been identified and will adjust the random sample locations as needed to achieve this. - Soil samples will also be collected at data gap locations or areas of suspected soil contamination. - Exposure areas, determined by current and reasonably foreseeable activities land uses, exposure routes, property ownership boundaries and topography.		- Supplemental analyses of soil samples obtained to fill in significant data gaps across the exposure area. - Exposure routes and receptors - Toxicological information on the contaminants of concern.
ii) Information Sources	- Existing soil/fill data - New results from all soil and fill samples collected from OU2, and data on background conditions. - Conceptual site model.		- New soil/fill data from the Phase 2 investigation - Available validated previous data (e.g., from Phase 1), within the exposure area.
iii) Basis of Action Level	Action Levels are: - USEPA Industrial and Residential Soil RSLs - USEPA RCRA ESLs The data collected will be compared against USEPA Residential and Industrial Soil RSLs and ESLs to identify potential human health and ecological risks associated with soil samples from OU2.		--
iv) Appropriate sampling & analysis methods	Methods are described in the Field Sampling Plan (CRA, May, 2013) and the Quality Assurance Project Plan (CRA, September 2008).		

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Residential and Industrial Soil Criteria***	***Comparison to Background Reference Conditions***	***Additional sampling (if necessary) to develop risk assessment exposure estimates***
Step 4: Define the Boundaries of the Study			
i) Target population, sample units	The initial target population is surficial and subsurface soils on the Southern Parcels. The sampling units are individual samples.	The sampling units are individual samples collected from the soil off-Site (beyond the Southern Parcels). The initial target population of background samples is surficial and subsurface soils from off-Site, nearby properties that have similar soil conditions to on-Site native soils.	Target population is soil and fill exceeding screening levels and comprising the exposure units for assessment of exposure risks for human receptors.
ii) Specify spatial boundaries	The spatial boundaries are the limits of site-related soil and fill contamination. Surficial soil is to a maximum depth of 2 ft bgs for human health risk purposes, and 3 ft bgs for ecological risk. The spatial boundaries of the sub-surface soil samples for screening human health risks will be to a depth of 15 ft bgs, i.e., the maximum soil depth construction workers would be expected to encounter. There is no predetermined maximum depth for characterizing the extent and magnitude of contamination. [Per the groundwater DQO in Worksheet #11-2, additional unsaturated soil samples will be collected at depths greater than 15 ft bgs to investigate potential leaching threats to groundwater.] Boreholes will be advanced a minimum of 5 ft into native material or until refusal, whichever is encountered first.	Background reference surface and subsurface sampling locations will be identified in areas outside a reasonable zone of potential influence (via surface runoff or substantial airborne dust deposition) for the Site. Distance from the Site and prevailing wind directions will be considered in making this determination.	The spatial boundaries are the limits of OU2, which is everywhere that environmental media have been impacted by Site contaminants outside of OU1. Surficial soil is to a maximum depth of 2 ft bgs for human health risk purposes, and 3 ft bgs for ecological risk. The spatial boundaries of the sub-surface soil samples for screening human health risks will be to a maximum depth of 15 ft bgs, i.e., the maximum soil depth construction workers would be expected to encounter. [Per the groundwater DQO in Worksheet #11-2, the spatial boundaries to evaluate risks to groundwater will be the entire depth of soil above the water table.]
iii) Specify temporal boundaries	The temporal boundaries are indefinite, assuming continued exposure at levels found during sampling. The practical temporal limits are based on the exposure assumptions of the Action Levels.		

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Residential and Industrial Soil Criteria***	***Comparison to Background Reference Conditions***	***Additional sampling (if necessary) to develop risk assessment exposure estimates***
Step 4: Define the Boundaries of the Study			
iv) Identify any other practical constraints	Practical constraints anticipated for sampling of OU2 soil and fill include the presence of cars on the Jim City Parcels and buildings and equipment on the Ron Barnett Parcels. Safety issues associated with sampling adjacent to surface water will also be considered for sampling activities on the Quarry Pond Parcels.	If different surficial soil substrates are encountered (e.g., silt vs. sand vs. clay), these differences may require additional sampling (e.g., further reference samples) to appropriately evaluate potential Site-related impacts. Off-Site sampling may be restricted by permission of property owners, and availability of suitable locations for background locations.	Practical constraints anticipated for sampling of Southern Parcels soil include the presence of cars on the Jim City Parcels and buildings and equipment on the Ron Barnett Parcels. Off-Site sampling, if required for delineation purposes, may be restricted by permission of property owners.
v.a) Scale of inference for decision making	Comparisons to Action Levels will be carried out on an individual-location basis.	Comparisons to background reference conditions will be carried out on an individual-location basis.	--
v.b) Scale of estimates			The scale of the exposure estimate is to be identified in a Site-specific risk assessment.
Step 5. Develop the Analytic Approach			
i.a) Specify Action Level	1) USEPA Industrial Soil RSLs 2) USEPA Residential Soil RSLs 3) USEPA RCRA ESLs	Background Threshold Values based on background reference data, following USEPA's ProUCL Technical Guide (2013)	--
i.b) Specify estimator	--		The arithmetic mean (per USEPA RAGS requirements) surface soil concentration of each contaminant that is greater than screening criteria.
ii.a) Specify population parameter of interest and theoretical decision rule	Individual observations at sampling locations on the Southern Parcels.		--
ii.b) Specify estimation procedure	--		

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Residential and Industrial Soil Criteria***	***Comparison to Background Reference Conditions***	***Additional sampling (if necessary) to develop risk assessment exposure estimates***
Step 6. Specify Performance or Acceptance Criteria			
i.a) Set baseline (null) and alternative hypotheses	Baseline H_0: soil sample concentrations are less than Action Levels. Alternative H_1: soil samples contain contaminant concentrations greater than Action Levels.	Baseline H_0: soil sample concentrations from the Southern Parcels are no different than reference background concentrations Alternative H_1: soil samples from the Southern Parcels contain contaminants at concentrations greater than reference conditions.	--
i.b) Specify how uncertainty accounted for in estimate	--		Uncertainty will be accounted for using a confidence interval on the population mean (per USEPA RAGS guidance).
ii.a) Determine impact of decision errors (false positives/negatives)	N/A: no statistical test is employed (direct / individual point-based comparison to Action Levels)	- If a false positive (Type I) error occurs, unnecessary additional investigation (Phase 2) may occur. - If a false negative (Type II) error occurs, conditions that are not due to background contaminant concentrations and pose potential health risks to receptors persist.	--
ii.b) Specify confidence level for estimate	--		The confidence level of the estimate will be 95 percent, unless specified otherwise (based on data distribution and/or the presence of non-detect results) in USEPA's ProUCL Technical Guide (2013).
iii) Specify "gray region" for test	N/A: no statistical test is employed (direct / individual point-based comparison to Action Levels)	N/A: since comparing individual concentrations against reference conditions, no statistical test is employed.	--
iv.a) Set tolerable limits on decision errors	N/A: no statistical test is employed (direct / individual point-based comparison to Action Levels)	The Background Threshold Values will be calculated using a 95 percent confidence level, making the false positive rate no greater than 5 percent. Limits on the false negative rate are not appropriate for comparisons of individual results to threshold values.	--

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Residential and Industrial Soil Criteria***	***Comparison to Background Reference Conditions***	***Additional sampling (if necessary) to develop risk assessment exposure estimates***
Step 6. Specify Performance or Acceptance Criteria			
iv.b) Specify performance or acceptance criteria	--		The lesser value of the 95 percent UCL on the population mean or the maximum individual measurement will be required.
Step 7: Develop the Plan for Obtaining Data			
i) Select sampling design	Soil samples from Southern Parcels will be collected from the exposure areas. Exposure areas are determined based on current use and ownership, potential future use, and topography. The exposure areas are defined in Section 5.2 of the OU2 RI/FS Work Plan.	Background surface and subsurface reference samples will be collected at 10 locations to provide a suitable data set (per USEPA's ProUCL Technical Guide, 2013) for the calculation of Background Threshold Values.	The number of additional soil samples required, for delineation purposes and removal of data gaps, will be determined based on the results of the Phase 1A and 1B investigations.
	Separate sets of data will be collected for (i) surface soil 0-2', (ii) subsurface soil 2-15', and (iii) unsaturated samples from a minimum of 24 locations at depths greater than 15 ft bgs. Additional soil samples will be collected at intervals within boreholes exhibiting evidence of contamination (based on field screening, visual and olfactory observations) A minimum of 8 samples per exposure area, per USEPA's ProUCL Technical Guide (2013), spaced on a regular grid with random origin (i.e., a systematic random sampling design), will be obtained for each exposure area identified in the risk assessment. Additional samples will be collected in the areas of any data gaps. Additional samples will be collected from subsurface soil (>15' at 3 locations per exposure area and additional locations) if impacts are identified.	--	--

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Residential and Industrial Soil Criteria***	***Comparison to Background Reference Conditions***	***Additional sampling (if necessary) to develop risk assessment exposure estimates***
Step 7: Develop the Plan for Obtaining Data			
ii) Specify/evaluate key assumptions supporting the design	The calculation of 95 percent upper confidence limits on a population mean makes assumptions of data characteristics (e.g., distribution and proportion of detected values), as fully discussed in the USEPA ProUCL Technical Guide (2013). Additionally, the presence of outlying values will be tested, and if present their impact on the values obtained evaluated.	The calculation of Background Threshold Values (statistical limits on an upper percentile, e.g., 95th) for the reference population of surficial soils depends on data characteristics (e.g., distribution and proportion of detected values), as fully discussed in the USEPA ProUCL Technical Guide (2013). Additionally, the presence of outlying values will be tested, and if present their impact on the values obtained evaluated.	The calculation of 95 percent upper confidence limits on a population mean makes assumptions of data characteristics (e.g., distribution and proportion of detected values), as fully discussed in the USEPA ProUCL Technical Guide (2013). Additionally, the presence of outlying values will be tested, and if present their impact on the values obtained evaluated.

Notes:

[1] If investigating a "decision problem", follow items ending in ".a" in subsequent DQO steps (e.g., "ii.a" or "iii.a")

If investigating an "estimation problem", follow ".b" items

Once the baseline risk assessment for OU2 has been performed, possible remedial goals (PRGs) will be derived from the calculator using site-specific risks

-- Item not applicable for the type of problem (decision vs. estimation) investigated

The planning team includes:
Respondents: Ken Brown (ITW); Jim Campbell (ITW); Bryan Heath (NCR); Wendell Barner (KELSEY HAYES CO.)
Steve Quigley (CRA project manager);

Wesley Dyck, Daniela Araujo (CRA statistics expert)

April Gowing, Steve Harris, Vincent Nero and Dan Smith (CRA risk assessment experts)

Rawa Fleisher, Angela Bown (CRA chemists/quality assurance staff)

Julian Hayward, Valerie Chan and Adam Loney (CRA project engineers); Alan Deal (CRA project hydro-geologists)

Leslie Patterson (USEPA Regional Project Manager); Madelyn Smith (Ohio EPA representative); and property owner stakeholders

11-2 - Groundwater Investigation

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Investigation of Soil/Fill on Southern Parcels***	***Comparison of Soil to Background***	***Groundwater Investigation (see OU1 Phase 2A/B DQO)***
Step 1. State the Problem			
i) Problem Description	Insufficient soil/fill quality data exist for OU2 in order to determine the presence or absence of risks to groundwater from contaminated soil or fill.	Insufficient groundwater quality data exist for OU2 in order to determine whether potential groundwater contamination is from the Site or from off-Site sources.	If soil/fill samples contain Site-related contaminant concentrations greater than USEPA SSL criteria for the protection of groundwater or Ohio EPA leach-based soil values, or if groundwater samples collected in the current (2013-2014) Phase 2A/B groundwater investigation contain Site-related contaminant concentrations greater than USEPA Maximum Contaminant Levels (MCLs) or Tapwater Regional Screening Level (RSL) criteria, a groundwater investigation will be conducted to delineate areas of OU2 groundwater contamination.
ii) Planning team	See note at bottom		
iii) Conceptual model	- Fill and/or contaminated soils above or below the water table may act as a source for groundwater contamination due to leaching and infiltration (Phase 1). Contaminated groundwater related to Site-activities may have migrated outside the boundaries of OU1. Shallow groundwater in the Upper Aquifer Zone typically flows westward and northward across the Site towards the Great Miami River (GMR), with a southwesterly component of flow oriented towards the Quarry Pond. Depending on the surface water elevation, it is apparent that groundwater in the Upper Aquifer Zone both discharges to, and is recharged by, the GMR. Thus, groundwater could transport contaminants to surface water. Groundwater flow in the Lower Aquifer Zone is predominantly southwest across the Site, with an occasional slight component of flow southeast towards monitoring wells MW-210B and MW-214. The lower aquifer is a designated sole-source aquifer. - VOCs, such as TCE, may volatilize from groundwater into vadose zone soil gas, which may migrate to indoor air via foundation cracks and utility penetrations in buildings, or may discharge to ambient air via dispersion (Phase 2). - The leachate seep investigation was completed over two days in September 2008. Field staff monitored the Site for the presence of leachate seeps through 2008 and 2009. No leachate seeps were observed during that time. Should leachate be observed during OU2 investigations, leachate seep sampling will be completed in accordance with the Leachate Seep Letter Work Plan (CRA, May 6, 2008).		
iv) General intended use for data	The soil data collected from each borehole will be used to identify areas in OU2 that may contribute to groundwater contamination. The data collected will be compared against Ohio EPA leach-based soil values and USEPA screening levels in soil (SSLs) that are protective of groundwater to identify risks associated with soil in OU2.		The OU1 Phase 2A/B data and any previously generated and validated data (historic monitoring wells and vertical aquifer samples (VAS)) will be used to determine the extent and magnitude of groundwater contamination above action levels, and generate exposure estimates for an assessment of ingestion, dermal contact, and inhalation of groundwater contaminants. The data will also be used to determine risks of groundwater contaminant volatilization into vadose zone soil gas, which may migrate to indoor air or discharge to ambient air. The data collected will ultimately be used in the Baseline Risk Assessment for OU2.

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Investigation of Soil/Fill on Southern Parcels***	***Comparison of Soil to Background***	***Groundwater Investigation (see OU1 Phase 2A/B DQO)***
Step 1. State the Problem			
v) Resources, constraints, deadlines	Sufficient resources will be committed to sample soil, groundwater, and leachate and seeps (if present) on the Southern Parcels and beyond (if necessary) under the OU2 RI/FS work plan. Sampling may be postponed due to flooding.		
Step 2. Goals of the Study			
i) Primary study question	Do soil samples from soil borings in OU2 contain Site-related contaminants at concentrations greater than Ohio EPA leach-based soil values or USEPA SSLs?		What is the extent of groundwater with Site-related contaminants exceeding USEPA MCLs, Tapwater RSLs, or USEPA Vapor Intrusion Screening Levels (VISLs)?
ii) Alternate outcomes or actions	- If sampling demonstrates that contaminant concentrations in soil are less than screening levels/criteria for leaching to groundwater, these potential migration pathways can be eliminated in the CSM for this area. - If soil samples collected from the boreholes demonstrate that contaminant concentrations in soils are greater than screening levels/criteria, and greater than background reference conditions, groundwater investigative activities may be warranted to identify and if necessary delineate groundwater plumes and/or fully characterize risks to human health.		- If sampling demonstrates that human health risks are acceptable, no further action is required. - If sampling demonstrates the presence of a Site-related groundwater contaminant plume, further study may be needed to evaluate alternatives for groundwater restoration. - If sampling demonstrates unacceptable human health risks, further evaluation, risk management and/or remediation would be required.
iii) Type of problem (decision or estimation) [1]	Decision (Action Level)		Decision (Action Level)
iv.a) Decision statement	Determine whether contaminant concentrations in the soil borings are greater than USEPA SSLs or Ohio EPA leach-based soil values.		Determine whether groundwater in OU2 with Site-related contamination poses an unacceptable ingestion, dermal contact, or inhalation risk to human health.
iv.b) Estimation statement & assumptions	--		--

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Investigation of Soil/Fill on Southern Parcels***	***Comparison of Soil to Background***	***Groundwater Investigation (see OU1 Phase 2A/B DQO)***
Step 3. Identify Information Inputs			
i) Information types needed	- Soil sample analysis from OU2 - Soil samples will be collected on a random basis (random oriented grid) across OU2. - Soil samples will also be collected at data gap locations (i.e. remaining geophysical anomalies) or areas of suspected soil contamination.	- Soil sample analysis from background locations	- Existing and newly-collected groundwater data from OU2.
ii) Information Sources	- Newly-collected and existing data from OU2	- Newly-collected and existing data from background locations.	- Newly-collected and validated data - Any available previous validated data (e.g., from historic monitoring wells and VAS samples) from OU2.
iii) Basis of Action Level	Action Levels are: - USEPA SSLs - Ohio EPA leach-based soil values		Action levels are: - USEPA MCLs, and RSLs for Tap Water where MCLs are unavailable - USEPA and / or ODH VISLs for groundwater
iv) Appropriate sampling & analysis methods	Methods are described in the Field Sampling Plan (CRA, May, 2013) and the Quality Assurance Project Plan (CRA, September 2008).		

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Investigation of Soil/Fill on Southern Parcels***	***Comparison of Soil to Background***	***Groundwater Investigation (see OU1 Phase 2A/B DQO)***
Step 4. Define the Boundaries of the Study			
i) Target population, sample units	- The target population is soil on the Southern Parcels, to be extended to soils elsewhere in OU2 if the extent of contamination above screening levels cannot be delineated in the Southern Parcels alone. The sampling units are individual samples collected from the soil.	- The target population is soil outside of OU1 and the Southern Parcels that are expected to represent background contaminant levels. The sampling units are individual samples collected from the soil.	Target population is groundwater within the Southern Parcels. If a Site-related groundwater plume extends beyond the Southern Parcels, additional sampling to delineate the plume will be necessary. Sampling units are individual groundwater samples collected from monitoring wells.
ii) Specify spatial boundaries	The spatial boundaries are the limits of Site-related contamination above screening levels. Additional unsaturated soil samples will be collected at depths greater than 15 ft bgs. Boreholes will be advanced through the entire thickness of fill material and up to approximately 5 feet into the underlying native material, or to 15 feet below ground surface (ft bgs), whichever occurs last.		The spatial boundaries are defined by the extent of Site-related groundwater contamination in OU2.
iii) Specify temporal boundaries	The temporal boundaries are indefinite, assuming continued exposure at levels found during sampling. The practical temporal limits are based on the exposure assumptions of the Action Levels.		- Permanent monitoring wells can be installed at any time based on the results of the soil/fill investigation. - Two sampling events total will be carried out at newly installed monitoring wells, during periods of high (i.e., February - April) or low (i.e., June - September) groundwater elevations. Seasonal groundwater flow fluctuations will be evaluated based on historic Site data, and will be demonstrated by the completion of a Site-wide groundwater elevation monitoring round completed prior to each sampling event.
iv) Identify any other practical constraints	- Practical constraints anticipated for sampling of Southern Parcel soil include the presence of cars on the Jim City Parcels and buildings and equipment on the Ron Barnett Parcels. - Safety issues associated with sampling adjacent to surface water will also be considered for sampling activities on the Quarry Pond Parcels.		
v.a) Scale of inference for decision making	Comparisons to Action Levels and background levels will be carried out on an individual-location basis.		
v.b) Scale of estimates	--		--

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Investigation of Soil/Fill on Southern Parcels***	***Comparison of Soil to Background***	***Groundwater Investigation (see OU1 Phase 2A/B DQO)***
Step 5. Develop the Analytic Approach			
i.a) Specify Action Level	Action Levels are: - USEPA SSLs - Ohio EPA leach-based soil values		Action levels are: - USEPA MCLs, and RSLs for Tap Water where MCLs are unavailable - USEPA VISLs and / or ODH for groundwater
i.b) Specify estimator	--		--
ii.a) Specify population parameter of interest and theoretical decision rule	Individual observations at sampling locations on the Southern Parcels, to be extended to soils elsewhere in OU2 if the extent of contamination above screening levels cannot be delineated in the Southern Parcels alone.		
ii.b) Specify estimation procedure	--		--
Step 6. Specify Performance of Acceptance Criteria			
i.a) Set baseline (null) and alternative hypotheses	Baseline H_0: soil sample concentrations are less than Action Levels Alternative H_1: soil samples contain contaminant concentrations greater than Action Levels		Baseline H_0: groundwater sample concentrations are less than Action Levels or are consistent with upgradient conditions (i.e., source is upgradient, either on or off-Site) Alternative H_1: groundwater sample concentrations are greater than Action Levels or upgradient conditions (i.e., contamination is related to Southern Site Parcels in OU2).
i.b) Specify how uncertainty accounted for in estimate	--		--
ii.a) Determine impact of decision errors (false positives/negatives)	N/A: no statistical test is employed (direct comparison to Action Levels)		N/A: no statistical test is employed (direct comparison to Action Levels)
ii.b) Specify confidence level for estimate	--		--
iii) Specify "gray region" for test	N/A: no statistical test is employed (direct comparison to Action Levels)		
iv.a) Set tolerable limits on decision errors	N/A: no statistical test is employed (direct comparison to Action Levels)		
iv.b) Specify performance or acceptance criteria	--		--

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Investigation of Soil/Fill on Southern Parcels***	***Comparison of Soil to Background***	***Groundwater Investigation (see OU1 Phase 2A/B DQO)***
Step 7. Develop the Plan for Obtaining Data			
i) Select sampling design	- Soil samples from Southern Parcels will be collected from each exposure area - Exposure areas are determined based on current use and ownership, potential future use, and topography. Exposure areas are detailed in Section 5.2 of the OU2 RI/FS Work Plan. - Separate sets of data will be collected for (i) surface soil 0-2', (ii) subsurface soil 2-15', and (iii) unsaturated samples from a minimum of 24 locations at depths greater than 15 ft bgs. - Additional soil samples will be collected at intervals within boreholes exhibiting evidence of contamination (based on field screening, visual and olfactory observations) - A minimum of 8 samples per exposure area, per USEPA's ProUCL Technical Guide (2013), spaced on a regular grid with random origin (i.e., a systematic random sampling design), will be obtained for each exposure area identified in the risk assessment. Additional samples will be collected in the areas of any data gaps (i.e. remaining geophysical anomalies). - Additional samples will be collected from subsurface soil (>15' at 3 locations per exposure area, and additional locations if impacts are identified).		- Groundwater samples from Southern Parcels will be collected from exposure areas with soil/fill concentrations greater than USEPA SSL criteria for the protection of groundwater or Ohio EPA leach-based soil values, or groundwater concentrations greater than USEPA RSLs from samples collected in the proposed (2014) Phase 1B/2A groundwater investigation - Exposure areas are determined based on current use and ownership, potential future use, and topography. Exposure areas are detailed in Section 5.2 of the OU2 RI/FS Work Plan - Monitoring wells will be installed at select locations identified as areas of potentially unacceptable risks or areas of significantly elevated contaminant concentrations. Respondents will discuss Phase 1 data, and all previous data with USEPA to determine the next steps and suitable locations of permanent monitoring wells. - Two sampling events will be carried out at newly installed monitoring wells. Parameters included in the second round of analysis may be decreased depending on the results of the first round.
ii) Specify/evaluate key assumptions supporting the design	The calculation of 95 percent upper confidence limits on a population mean makes assumptions of data characteristics (e.g., distribution and proportion of detected values), as fully discussed in the USEPA ProUCL Technical Guide (2013). Additionally, the presence of outlying values will be tested, and if present their impact on the values obtained evaluated.		

Notes:

[1] If investigating a "decision problem", follow items ending in ".a" in subsequent DQO steps (e.g., "ii.a" or "iii.a").

If investigating an "estimation problem", follow ".b" items.

Once the baseline risk assessment for OU2 has been performed, possible remedial goals (PRGs) will be derived from the calculator using site-specific risks

-- Item not applicable for the type of problem (decision vs. estimation) investigated.

The planning team includes:
Respondents: Ken Brown (ITW); Jim Campbell (ITW); Bryan Heath (NCR); Wendell Barner (KELSEY HAYES CO.)
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Leslie Patterson (USEPA Regional Project Manager); Madelyn Smith (Ohio EPA representative); and property owner stakeholders.

11-3 - Soil Gas Investigation

Investigative Phase:	Phase 1	Phase 2
Investigative Item:	***Investigation of Soil, Fill and Groundwater***	***Soil Gas Probe Investigation based on Southern Parcels Soil, Fill and Groundwater Investigations (if necessary)***
Step 1. State the Problem		
i) Problem description	- The OU2 Southern Site Parcel soil and fill areas have not been fully characterized, and they may contain materials that can produce elevated concentrations of explosive gases and NMOCs in landfill gas, and VOCs in soil gas. - Businesses operating on Site are located above or immediately adjacent to fill material, in close proximity to the soil gas probe locations where elevated levels of VOCs and explosive gases were detected. - A data gap exists with respect to possible groundwater contamination outside of OU1 that may have concentrations capable of posing a vapor intrusion threat. - A data gap exists with respect to potential soil contamination that may pose a vapor intrusion threat to businesses operating on or near the Southern Parcels.	- If soil, fill, or groundwater samples containing Site-related contaminant concentrations with the potential to produce landfill gas/soil vapor are identified, actual soil gas concentrations will be investigated through the installation of soil gas probes in the affected area to assess the present conditions and potential for migration.
ii) Planning team	See note at bottom	
iii) Conceptual model	- VOCs, such as TCE, may volatilize from groundwater, soil, or subsurface landfill contents into vadose zone soil gas, which may migrate to indoor air via foundation cracks and utility penetrations in buildings. - Workers or residents in buildings where VOCs are present at concentrations greater than target criteria may be subject to potential risks due to inhalation hazards. - Potential future users of the Site include workers both outdoors and in buildings on areas of the site that are currently vacant.	
iv) General intended use for data	The collected soil/fill and groundwater data will be used to evaluate the potential for soil, fill, or groundwater contamination to act as a source for landfill gas/soil vapor, and to identify areas with potential landfill gas/soil vapor impacts.	The collected soil gas data will be used for direct comparison to the action levels, and each result will represent a reasonable worst-case maximum potential concentration migrating to indoor air at each structure. The data collected will ultimately be used in the Baseline Risk Assessment for OU2.
v) Resources, constraints, deadlines	An iterative sampling approach may be required to refine estimates based on earlier findings from the OU1 vapor intrusion investigation.	Sufficient resources have been reserved to collect and analyze soil gas from the probes. Sampling may be constrained by access agreements to off-Site parcels or buildings. An iterative sampling approach may be required to refine estimates based on findings from the soil, fill, and groundwater investigations.

Investigative Phase:	Phase 1	Phase 2
Investigative Item:	***Investigation of Soil, Fill and Groundwater***	***Soil Gas Probe Investigation based on Southern Parcels Soil, Fill and Groundwater Investigations (if necessary)***
Step 2. Goals of the Study		
i) Primary study question	Does OU2 soil, fill, or groundwater contain Site-related contaminant concentrations that indicate VOCs or methane in soil gas may pose a threat to human health?	- Do contaminant concentrations in soil vapor pose an unacceptable risk, via the vapor intrusion pathway, to occupants of structures on or immediately adjacent to the Site? - Are concentrations of combustible gases within a structure greater than the screening criteria of 1 and 10 percent of the LEL (as per the USEPA Region V Vapor Intrusion Guidebook, October 2010), or the regulatory criterion of 25 percent of the LEL (as per OAC Chapter 3745-27-12)? - Taken together, how do the concentrations of contaminants and combustible gases in soil vapor affect future use of the Site? - Does the OU2 soil vapor act as a source of soil gas to the structures studied in the Vapor Intrusion investigation?
ii) Alternate outcomes or actions	- If soil/fill borehole samples and/or groundwater samples contain VOCs at concentrations less than the action levels, and methane below 1 and 10 percent of the LEL, no further action is necessary. - If VOCs and/or methane are present at concentrations greater than the action levels and 1 and 10 percent of the LEL, then further evaluation is required.	- If soil gas samples contain VOCs at concentrations less than the action levels, and methane below 1 and 10 percent of the LEL, no further action is necessary. - If VOCs and/or methane are present at concentrations greater than the action levels and 1 and 10 percent of the LEL, then further evaluation is required.
iii) Type of problem (decision or estimation)[1]	Decision (Action Level)	Decision (Action Level)
iv.a) Decision statement	Determine whether VOCs are present in OU2 soil/fill material and groundwater at levels posing a potential risk to occupants of current and future on-Site structures.	Determine whether VOCs are present in the OU2 areas at levels posing potential risk to potential current and future occupants of off-Site structures identified as being at risk from volatilization of groundwater into indoor air based on and OU2 soil investigation and Phase 2 of the Groundwater DQO investigation.
iv.b) Estimation statement & assumptions	--	--

Investigative Phase:	Phase 1	Phase 2
Investigative Item:	***Investigation of Soil, Fill and Groundwater***	***Soil Gas Probe Investigation based on Southern Parcels Soil, Fill and Groundwater Investigations (if necessary)***
Step 3. Identify Information Inputs		
i) Information types needed	- Analytical data from soil boreholes installed within the soil and fill material, and monitoring well groundwater samples.	- This would be a new data collection effort, with analyses performed on samples collected from soil gas probes installed within the soil and/or fill material.
ii) Information Sources	- New data from the OU2 soil and groundwater investigations will form the basis of assessment.	- New data from the OU2 soil vapor/landfill gas investigation will form the basis of assessment.
iii) Basis of Action Level	Action Levels are: - Ohio Department of Health (ODH) Industrial and Residential Action Levels -USEPA Vapor Intrusion Screening Levels (VISLs: groundwater, indoor air, and sub-slab soil vapor levels calculated from USEPA RSLs and ODH screening levels for air inhalation). - 1, 10, or 25 percent of the LEL	
iv) Appropriate sampling & analysis methods	- Methods are described in the Field Sampling Plan (CRA, May, 2013) and the Quality Assurance Project Plan (CRA, September 2008). - During the soil borehole investigation, methane values will be recorded in the field using a Landtec GEM-2000, or equivalent equipped with a charcoal carbon filter to differentiate methane from VOCs.	- Methods are described in the Vapor Intrusion Investigation Work Plan (USEPA, November 2011) and Field Sampling Plan (CRA, May, 2013). VOC and naphthalene analysis is via EPA method TO-15. - During soil gas probe installation, methane values will be recorded in the field using a Landtec GEM-2000, or equivalent.

Investigative Phase:	Phase 1	Phase 2
Investigative Item:	***Investigation of Soil, Fill and Groundwater***	***Soil Gas Probe Investigation based on Southern Parcels Soil, Fill and Groundwater Investigations (if necessary)***
Step 4. Define the Boundaries of the Study		
i) Target population, sample units	The target population is surficial and subsurface soils and fill, and groundwater on the Southern Parcels (and beyond the Southern Parcels, if necessary). The sampling units are individual samples collected from the soil, divided into background reference, and exposure units for assessment of risks to human receptors.	Target population is soil gas within the soils and/or the fill area where potential VOC-containing residues are present in the vadose zone, or concentrations of VOCs in groundwater are greater than Phase 1 action levels, and therefore, represent a vapor intrusion risk.
ii) Specify spatial boundaries	Spatial boundaries are initially the limits of the Southern Parcels within the OU2 boundary, which included the fill area and occupied buildings.	Spatial boundaries are (initially) the limits of the Southern Parcels within the OU2 boundary, which includes the fill area and occupied buildings, where VOC residues are present in the vadose zone or concentrations of contaminants in groundwater are greater than Phase 1 Action Levels. If soil vapor/landfill gas migration beyond the Southern Parcels is indicated by either Phase 1 or Phase 2 sampling, additional soil probes outside of the Southern Parcels will be necessary.
iii) Specify temporal boundaries	The temporal boundaries are indefinite, assuming continued exposure at levels found during sampling. The practical temporal limits are based on exposure assumptions used in the derivation of the Action Levels.	
iv) Identify any other practical constraints	- Practical constraints anticipated for sampling of Southern Parcel soil and fill include the presence of cars on the Jim City Parcels and buildings and equipment on the Ron Barnett Parcels. - Safety issues associated with sampling adjacent to surface water will also be considered for sampling activities on the Quarry Pond Parcels.	- Practical constraints anticipated for sampling of Southern Parcel soil gas include the presence of cars on the Jim City Parcels and buildings and equipment on the Ron Barnett Parcels. - Safety issues associated with sampling adjacent to surface water will also be considered for sampling activities on the Quarry Pond Parcels. - Depending on soil borehole sample analytical results, the soil gas probe may not be able to be screened in intervals that delineate the specific stratigraphic layer(s) contributing to combustible gas concentrations.
v.a) Scale of inference for decision making	The initial decision unit is the soil, fill, and groundwater within the Southern Parcels. The decision unit may be expanded to soil, fill, and groundwater beyond the Southern Parcels, if necessary.	The initial decision unit is the soil gas within the Southern Parcels. The decision unit may be expanded to soil gas beyond the Southern Parcels, if necessary.
v.b) Scale of estimates	--	

Investigative Phase:	Phase 1	Phase 2
Investigative Item:	***Investigation of Soil, Fill and Groundwater***	***Soil Gas Probe Investigation based on Southern Parcels Soil, Fill and Groundwater Investigations (if necessary)***
Step 5. Develop the Analytic Approach		
i.a) Specify Action Level	1) USEPA Industrial Soil RSLs for Inhalation Screening Levels 2) USEPA Residential Soil RSLs for Inhalation Screening Levels	1) ODH Industrial and Residential Action Levels 2) USEPA Vapor Intrusion Screening Levels (VISLs: groundwater, indoor air, and sub-slab air levels calculated from USEPA RSLs for air inhalation). 3) 1 and 10 percent of the LEL 4) 25 percent of the LEL
i.b) Specify estimator	--	
ii.a) Specify population parameter of interest and theoretical decision rule	Individual observations at sampling locations on the Southern Parcels	Maximum concentration in soil gas samples and explosive gas measurements at each structure compared directly to criteria.
ii.b) Specify estimation procedure	--	
Step 6. Specify Performance or Acceptance Criteria		
i.a) Set baseline (null) and alternative hypotheses	Baseline H_0: soil or groundwater contamination concentrations are less than Action Levels Alternative H_1: soil or groundwater contamination concentrations are greater than Action Levels	Baseline H_0: soil vapor contamination concentrations are less than Action Levels Alternative H_1: soil vapor contamination concentrations are greater than Action Levels
i.b) Specify how uncertainty accounted for in estimate	--	
ii.a) Determine impact of decision errors (false positives/negatives)	N/A: since comparing to maximum value, no statistical test is employed	
ii.b) Specify confidence level for estimate	--	
iii) Specify "gray region" for test	N/A: since comparing to maximum value, no statistical test is employed	
iv.a) Set tolerable limits on decision errors	N/A: since comparing to maximum value, no statistical test is employed	
iv.b) Specify performance or acceptance criteria	--	

Investigative Phase:	Phase 1	Phase 2
Investigative Item:	***Investigation of Soil, Fill and Groundwater***	***Soil Gas Probe Investigation based on Southern Parcels Soil, Fill and Groundwater Investigations (if necessary)***
Step 7. Develop the Plan for Obtaining Data		
i) Select sampling design	See Step 7i) of QAPP Worksheets 11-1 and 11-2	- CRA will install temporary soil gas probes at select locations dependent on the observations CRA makes during the drilling of the soil borings - CRA will assess the need for further soil gas or vapor intrusion monitoring within or beyond the fill material limits, based on the results of the initial monitoring.
ii) Specify/evaluate key assumptions supporting the design	--	

Notes:

[1] If investigating a "decision problem", follow items ending in ".a" in subsequent DQO steps (e.g., "ii.a" or "iii.a").

If investigating an "estimation problem", follow ".b" items.

Once the baseline risk assessment for OU2 has been performed, possible remedial goals (PRGs) will be derived from the calculator using site-specific risks.

-- Item not applicable for the type of problem (decision vs. estimation) investigated.

The planning team includes:
Respondents: Ken Brown (ITW); Jim Campbell (ITW); Bryan Heath (NCR); Wendell Barner (KELSEY HAYES CO.)
Steve Quigley (CRA project manager);

Wesley Dyck, Daniela Araujo (CRA statistics expert);

April Gowing, Steve Harris, Vincent Nero and Dan Smith (CRA risk assessment experts);

Rawa Fleisher, Angela Bown (CRA chemists/quality assurance staff);

Julian Hayward, Valerie Chan and Adam Loney (CRA project engineers); Alan Deal (CRA project hydro-geologist);

Leslie Patterson (USEPA Regional Project Manager); Madelyn Smith (Ohio EPA representative); and property owner stakeholders.

11-4 - Surface Water

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Ambient Water Quality Criteria***	***Comparison to Upstream Conditions***	***Quarry Pond Surface Water Sampling***
Step 1. State the Problem			
i) Problem description	Surface water samples have not previously been obtained from the Great Miami River (GMR) as it flows past by the Site. It is unknown whether and to what extent the Site has any measurable impact on water quality in the GMR. Intermittent drainage pathways and leachate seeps have not been identified at the Site to date.		Limited historic surface water samples have been obtained from the Quarry Pond. Historic Quarry Pond surface water samples did not contain any VOCs. No other parameters were assessed. The impact of Site contaminants on the Quarry Pond is not known. Intermittent drainage pathways have not been identified at the Site to date.
ii) Planning team	See note at bottom		
iii) Conceptual model	- Shallow groundwater from the Site typically flows towards the west and/or north towards the GMR, which could carry contaminants into its surface waters. - Erosion of surface soils from the Site could also carry Site-related contaminants to the GMR, which is at a lower elevation, via overland surface flow. - During flood events, any potential GMR contaminants originating off-Site could affect the Site. - Greater contaminant concentrations may be present at groundwater discharge points into the GMR and this will be investigated through sampling completed along transects. - Persons can come into contact with river water when using the GMR for recreation. - Wildlife and aquatic organisms are in contact with and ingest GMR water.		- Shallow and deep groundwater from the Site typically flows towards the west towards the Quarry Pond, which could carry contaminants into the Quarry Pond. - During flood events, off-Site contaminants could be deposited in the Quarry Pond. - Erosion of surface soils from the Site could also carry Site-related contaminants to the Quarry Pond, which is at a lower elevation, via overland surface flow. - Persons can come into contact with pond water when using the pond area for recreation. - Wildlife and aquatic organisms are in contact with and ingest Quarry Pond water.
iv) General intended use for data	The data collected will be compared against ambient water quality criteria to assess if human or aquatic ecosystem health is potentially impaired. In addition, CRA will visually inspect the bank of the GMR adjacent to the Site for evidence of leachate and/or runoff discharges potentially related to the Site (i.e., erosion rills, iron oxidation, turbidity, etc.). Sample locations will be matched up with Site discharges, if observed. The data collected will ultimately be used in the Baseline Risk Assessment for OU2.	The data collected from sampling locations along the Site's boundaries will be compared to upstream (background) conditions, to determine if there are any measurable inputs of contaminants from the Site. The data collected will ultimately be used in the Baseline Risk Assessment for OU2.	The data collected will be compared against ambient water quality criteria to assess if human health or aquatic ecosystem health is potentially impaired. In addition, CRA will visually inspect the Quarry Pond embankments for evidence of leachate and/or runoff discharges (i.e., erosion rills, iron oxidation, turbidity, etc.). Sample locations will be matched up with Site discharges, if observed. The data collected will ultimately be used in the Baseline Risk Assessment for OU2.

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Ambient Water Quality Criteria***	***Comparison to Upstream Conditions***	***Quarry Pond Surface Water Sampling***
Step 1. State the Problem			
v) Resources, constraints, deadlines	Surface water quality and storm water runoff may be influenced by rainfall events, water temperature and other seasonal effects, which requires monitoring at different times of the year and under different conditions. Surface water sampling may not be possible during high flows. Surface water and storm water runoff sampling may not be possible during ice-cover conditions. Surface water sampling will be completed during low flow periods where contaminants entering via groundwater would present the greatest risks. Storm water runoff sampling will be completed following rainfall events should a significant runoff pathway be identified. Intermittent drainage pathways have not been identified at the Site to date.		
Step 2. Goals of the Study			
i) Primary study question	Does surface water quality fail to meet ambient water quality criteria for protection of human health (direct contact, ingestion, and ingestion of aquatic organisms), and aquatic organisms?	Does the Site add contaminants to surface water in the GMR as it flows past the Site? If so, to what extent?	Does surface water quality fail to meet ambient water quality criteria for protection of aquatic organisms and human health (trespassers, recreational users and anglers)?
ii) Alternate outcomes or actions	- If sampling demonstrates that ambient water quality criteria are met, no further monitoring is planned. - If sampling demonstrates that criteria are not met, comparison with background conditions is warranted.	- If sampling demonstrates conditions adjacent to the Site are less than or equal to those found upstream, no further monitoring is planned. - If sampling demonstrates conditions are greater than upstream, and that contaminant concentrations are greater than Action Level criteria (see Phase 1A to left), further evaluation and/or control measures may be warranted.	- If sampling demonstrates that ambient water quality criteria are met, no further monitoring is planned. - If sampling demonstrates that criteria are not met, further evaluation and/or control measures may be warranted.
iii) Type of problem (decision or estimation)[1]	Decision (Action Level)		
iv.a) Decision statement	Determine whether any contaminants are present at concentration greater than ambient water quality criteria in the GMR as it flows past the Site.	Determine whether any measurable input of contaminants from the Site, relative to upstream conditions, occurs in the GMR as it flows past the Site.	Determine whether any contaminants are greater than ambient water quality criteria in the Quarry Pond.
iv.b) Estimation statement & assumptions	--		

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Ambient Water Quality Criteria***	***Comparison to Upstream Conditions***	***Quarry Pond Surface Water Sampling***
Step 3. Identify Information Inputs			
i) Information types needed	Surface water sample analysis is required to assess conditions in the GMR as it flows past the Site.		Surface water samples are required to assess conditions in the Quarry Pond.
ii) Information Sources	New data from the investigation will form the basis of assessment.		New data from the investigation will form the basis of assessment.
iii) Basis of Action Level	Action Levels are: - Ambient water quality criteria (Ohio drainage basin) - Ohio EPA Aquatic Life and Human Health Tier I and II Values - USEPA RSL (tapwater) - USEPA National Recommended Water Quality Criteria for human health for consumption of water + organisms	The selected Action Level is a Background Threshold Value (e.g., 95th percentile) based on upstream conditions.	Action Levels are: - Ambient water quality criteria (Ohio drainage basin) - Ohio EPA Aquatic Life and Human Health Tier I and II Values - USEPA RSL (tapwater) - USEPA National Recommended Water Quality Criteria for human health for consumption of water + organisms
iv) Appropriate sampling & analysis methods	Methods are described in the Field Sampling Plan (CRA, May, 2013), CRA's Standard Operating Procedures, and the Quality Assurance Project Plan (CRA, September 2008). VOC samples will be collected using a peristaltic pump to minimize sample aeration while allowing for sample preservation. All other parameters will be sampled by directly dipping sample containers in the surface water body (GMR or Quarry Pond).		

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Ambient Water Quality Criteria***	***Comparison to Upstream Conditions***	***Quarry Pond Surface Water Sampling***
Step 4. Define the Boundaries of the Study			
i) Target population, sample units	The target population is all water flowing in the GMR as it flows past the Site. The sampling units are individual grab samples collected from the GMR, divided into upstream and near-Site reaches. The surface water sample locations will be adjusted based on the location of intermittent drainage pathways and GMR discharge points, if any are identified.		The target population is all water in the Quarry Pond. The sampling units are individual grab samples collected from the Quarry Pond. The surface water sample locations will be adjusted based on the location of intermittent drainage pathways and GMR discharge points, if any are identified.
ii) Specify spatial boundaries	In order to ensure that any potential contributions from nearby facilities (e.g., former GM-Delphi plant) are accounted for, CRA proposes to specify upstream sampling locations as those occurring to the east of Dryden Road, on the near-Site side of any dams. Near-Site sampling locations are those occurring to the west of Dryden Road (i.e., as surface water flows past the Site), and these will be located on the near (south/east) shore of the GMR. Due to the industrial activity in the area, chemical use and contaminants in the area may have been used by more than one facility. In order to establish whether contamination is or has resulted from Site activities, the background locations have been set close to the Site.		Spatial boundaries are the boundaries of Quarry Pond surface water.
iii) Specify temporal boundaries	The temporal boundaries are defined by the duration of monitoring, which will occur over two sampling rounds		The temporal boundaries are defined by the duration of monitoring, which will occur over two sampling rounds.
iv) Identify any other practical constraints	Sampling may be postponed due to flooding or ice conditions in the GMR. The outfall of the City of Dayton Waste Water Treatment Plant across the river GMR, just south of the downstream limit of the Site, may substantially impact downstream water quality, making any subsequent Site effects difficult to discern. If any dams/weirs are encountered, samples will be collected from the side of the dam closest to the Site (i.e., downstream of any upstream dams, and upstream of any downstream dams). Dilution of contaminants is likely towards the center and far bank of the GMR, and increases with distance downstream of the Site.		Sampling may be postponed due to flooding or ice conditions in the Quarry Pond.
v.a) Scale of inference for decision making	Comparisons to Action Levels will be carried out on an individual-location basis. For the RA, the 95 percent UCL of the mean concentration in an exposure unit will be used. A single exposure unit will be applied for the GMR.	Comparisons to upstream conditions will be carried out on an individual-location basis.	Comparisons to Action Levels will be carried out on an individual-location basis.
v.b) Scale of estimates	--		

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Ambient Water Quality Criteria***	***Comparison to Upstream Conditions***	***Quarry Pond Surface Water Sampling***
Step 5. Develop the Analytic Approach			
i.a) Specify Action Level	- Ambient Water Quality Criteria - Ohio EPA Aquatic Life and Human Health Tier I and II Values - USEPA RSL (tapwater) - USEPA National Recommended Water Quality Criteria for human health for consumption of water + organisms		Background Threshold Values based on upstream data, following USEPA's ProUCL Technical Guide (2013)
i.b) Specify estimator	--		
ii.a) Specify population parameter of interest and theoretical decision rule	Individual observations at near-Site sampling locations.		--
ii.b) Specify estimation procedure	--		--

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Ambient Water Quality Criteria***	***Comparison to Upstream Conditions***	***Quarry Pond Surface Water Sampling***
Step 6. Specify Performance or Acceptance Criteria			
i.a) Set baseline (null) and alternative hypotheses	Baseline H_0: surface water concentrations are less than Action Levels Alternative H_1: surface water concentrations are greater than Action Levels	Baseline H_0: near-Site surface water is no different than upstream Alternative H_1: near-Site surface water contains contaminant concentrations greater than upstream conditions	Baseline H_0: surface water concentrations are less than Action Levels Alternative H_1: surface water contaminant concentrations are greater than Action Levels
i.b) Specify how uncertainty accounted for in estimate	--		
ii.a) Determine impact of decision errors (false positives/negatives)	N/A: no statistical test is employed (direct comparison to Action Levels)	- If a false positive (Type I) error occurs, unnecessary additional investigation may occur. - If a false negative (Type II) error occurs, conditions that are not due to background conditions and that pose potential risk to aquatic ecosystem and/or human receptors could persist.	N/A: no statistical test is employed (direct comparison to Action Levels)
ii.b) Specify confidence level for estimate	--		
iii) Specify "gray region" for test	N/A: no statistical test is employed (direct comparison to Action Levels)	N/A: since comparing to maximum value, no statistical test is employed	N/A: no statistical test is employed (direct comparison to Action Levels)
iv.a) Set tolerable limits on decision errors	N/A: no statistical test is employed (direct comparison to Action Levels)	The Background Threshold Values will be calculated using a 95 percent confidence level, making the false positive rate no greater than 5 percent. Since individual near-Site samples will be compared against background samples, the false negative rate will be controlled by two sampling events completed over the study period. An assessment of the decision performance curve achieved based on the monitoring data will be undertaken.	N/A: no statistical test is employed (direct comparison to Action Levels)
iv.b) Specify performance or acceptance criteria	--		

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Ambient Water Quality Criteria***	***Comparison to Upstream Conditions***	***Quarry Pond Surface Water Sampling***
Step 7. Develop the Plan for Obtaining Data			
i) Select sampling design	Near-Site samples will be collected close to the proximate (south/east) shore of the GMR, at the mid-point of the GMR at the upstream edge of the Site, and on the near-Site side of any dams; and at intervals of 800 ft (12 samples per event). Prior to surface water sample collection, a Site boundary visual inspection will be completed to identify any areas of discharge (i.e., rust stains, eddies, sediment, etc.) Surface water sampling will be completed during periods of GMR low-flow and the two sampling rounds will be completed at least three months apart.	Upstream samples will be collected at different locations, on the near-Site side of any dams, to provide a suitable data set (8-10 samples, per USEPA's ProUCL Technical Guide, 2013) for the calculation of Background Threshold Values. Near-Site samples will be collected along two three-point transects, upstream of the Site. Surface water sampling will be collected during periods of GMR low-flow and the two sampling rounds will be completed at least three months apart.	Prior to surface water sample collection, visual inspection of the Quarry Pond embankment will be completed to identify any areas of discharge (i.e., rust stains, eddies, sediment, etc.). Five samples will be collected at various points within the Quarry Pond in each of two sampling events (10 samples total). Two sampling rounds will be completed at least three months apart.
ii) Specify/evaluate key assumptions supporting the design	Mixing in the GMR is expected to be reasonably complete over the travel length of the GMR (greater than one mile) adjacent to the Site. Sampling at key locations (upstream edge, mid-Site, upstream of the WWTP, and downstream) will represent the range of ambient conditions in surface water.	The calculation of Background Threshold Values (statistical limits on an upper percentile, e.g. 95th) for the upstream population of surface waters depends on data characteristics (e.g., distribution and proportion of detected values), as fully discussed in the USEPA ProUCL Technical Guide (2013). Additionally, the presence of outlying values will be tested, and if present their impact on the values obtained evaluated.	--

Notes:

[1] If investigating a "decision problem", follow items ending in ".a" in subsequent DQO steps (e.g., "ii.a" or "iii.a").

If investigating an "estimation problem", follow ".b" items.

Once the baseline risk assessment for OU2 has been performed, possible remedial goals (PRGs) will be derived from the calculator using site-specific risks.

-- Item not applicable for the type of problem (decision vs. estimation) investigated.

The planning team includes:
Respondents: Ken Brown (ITW); Jim Campbell (ITW); Bryan Heath (NCR); Wendell Barner (KELSEY HAYES CO.)
Steve Quigley (CRA project manager);

Wesley Dyck, Daniela Araujo (CRA statistics expert);

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Leslie Patterson (USEPA Regional Project Manager); Madelyn Smith (Ohio EPA representative); and property owner stakeholders.

11-5 - Sediment

Medium:	Great Miami River (GMR) Sediment			Quarry Pond Sediments
Investigation Phase:	**Phase 1A – GMR**	**Phase 1B – GMR**	**Phase 2 – GMR**	**Phase 1A – Quarry Pond**
Investigation Item:	***Comparison to Human Health and Ecological Screening Values***	***Comparison to Upstream Conditions***	***Benthic Sampling***	***Comparison to Human Health and Ecological Risk Screening Values***
Step 1. State the Problem				
i) Problem description	It is unknown whether the Site has a measurable impact on sediment quality in the Great Miami River (GMR). Previous (GMR) sampling found polycyclic aromatic hydrocarbon (PAH) concentrations and some pesticide concentrations greater than conservative Ecological Screening Levels (ESLs), and arsenic and PAHs concentrations greater than USEPA Residential Soil RSLs. However, these common contaminants were also found, in similar concentrations, in upstream samples taken by OEPA (1995) in routine sampling of the GMR. Therefore, further data are needed to assess whether downstream concentrations are greater than upstream concentrations and, if so, whether downstream samples pose potential risks to ecological and human receptors.		If contaminant concentrations are greater than sediment benchmarks protective of aquatic life (Phase 1A-GMR), significantly greater than upstream concentrations (Phase 1B-GMR), and are potentially Site-related, a benthic community survey will be completed in accordance with USEPA Rapid Bio assessment Protocols (EPA 841-B-99-002) or OEPA assessment methods.	Previous Quarry Pond sediment sampling found PAH concentrations greater than conservative ESLs, and arsenic and PAH concentrations greater than USEPA Industrial Soil RSLs. Further data are needed to assess the magnitude and extent of Quarry Pond sediment contamination and, whether Quarry Pond sediments pose potential risks to ecological and human health risks.
ii) Planning team	See note at bottom			
iii) Conceptual model	- Shallow groundwater from the Site typically flows towards the west and/or north towards the GMR, which could carry contaminants into its sediment. - Contaminants in sediment can be toxic to benthic organisms. -Fish may uptake contaminants in sediments and can be eaten by other fish, birds, and humans.			- Shallow and deep groundwater from the Site typically flows towards the west towards the QP, which could carry contaminants into its sediment. - PAH concentrations greater than conservative ESLs, and arsenic and PAH concentrations greater than USEPA Industrial Soil RSLs, have been found in Quarry Pond sediment.
	- Erosion of surface soils from the Site could also carry Site-related contaminants to the GMR and/or the Quarry Pond, which is at a lower elevation, via overland surface flow. - During flood events, off-site contaminants could be deposited on-site. - Contaminants could be toxic to benthic organisms and impact other species in the aquatic ecosystem. - Persons use the GMR and Quarry Pond for recreation, mainly in boats; however, they could come into dermal contact with the sediment. - Persons consume the fish caught in the Quarry Pond.			

Medium:	Great Miami River (GMR) Sediment			Quarry Pond Sediments
Investigation Phase:	**Phase 1A – GMR**	**Phase 1B – GMR**	**Phase 2 – GMR**	**Phase 1A – Quarry Pond**
Investigation Item:	***Comparison to Human Health and Ecological Screening Values***	***Comparison to Upstream Conditions***	***Benthic Sampling***	***Comparison to Human Health and Ecological Risk Screening Values***
Step 1. State the Problem				
iv) General intended use for data	The sediment data collected will be compared against ESLs to assess whether aquatic ecosystem health is potentially impaired. The sediment data will be used to determine if bioaccumulative contaminants are present and to model edible fish concentrations for the HHRA. Additionally, CRA will compare the data to USEPA Industrial Soil RSLs as a screening evaluation to identify potential human health risks. Industrial Soil RSLs are proposed as a surrogate for human exposure risks from sediments, due to the limited exposure frequency in the GMR compared to a residential exposure scenario. Residential Soil RSLs will be used as an initial screening step to account for early-life susceptibility to mutagens for child receptors. The data collected will ultimately be used in the Baseline Risk Assessment for OU2.	The data collected from sampling locations adjacent to the landfill's boundaries will be compared to upstream conditions, to determine if there are any measurable inputs of contaminants from the Site. The data collected will ultimately be used in the Baseline Risk Assessment for OU2.	The data collected will be used to detect aquatic life impairments and assess their relative severity. The data collected will ultimately be used in the Baseline Risk Assessment for OU2.	The data collected will be compared against ESLs to assess if Quarry Pond aquatic ecosystem health is potentially impaired. Additionally, CRA will compare the data to USEPA Industrial Soil criteria to identify any potential human health risks. Industrial Soil RSLs are proposed as a surrogate for human exposure risks from sediments, due to the limited exposure frequency in the Quarry Pond compared to a residential exposure scenario. Residential Soil RSLs will be used as an initial screening step to account for early-life susceptibility to mutagens for child receptors. The data collected will ultimately be used in the Baseline Risk Assessment for OU2. The data will be used to determine if there is a need to cap or otherwise remediate the sediments in the Quarry Pond. The sediment data will be used to determine if bioaccumulative contaminants are present and to model edible fish concentrations for the HHRA.

Medium:	**Great Miami River (GMR) Sediment**			**Quarry Pond Sediments**
Investigation Phase:	**Phase 1A – GMR**	**Phase 1B – GMR**	**Phase 2 – GMR**	**Phase 1A – Quarry Pond**
Investigation Item:	***Comparison to Human Health and Ecological Screening Values***	***Comparison to Upstream Conditions***	***Benthic Sampling***	***Comparison to Human Health and Ecological Risk Screening Values***
v) Resources, constraints, deadlines	Sufficient resources will be committed to sample sediments under the OU2 RI/FS work plan.			Sufficient resources will be committed to sample sediments under the OU2 RI/FS work plan.
Step 2. Goals of the Study				
i) Primary study question	Does sediment in the GMR and/or Quarry Pond contain Site-related contaminants at concentrations greater than ESLs and/or Industrial Soil RSLs and/or Residential Soil RSLs for protection of human health?	Does the Site add significantly to contaminants in sediments in the GMR adjacent to and down-gradient of the Site?	Are benthic organisms at risk due to sediment concentrations caused by Site-related contamination?	Do sediments in the Quarry Pond contain contaminant concentrations greater than ESLs and/or Industrial Soil RSLs for protection of human health?
ii) Alternate outcomes or actions	- If sampling demonstrates that contaminants in sediment are less than screening levels/criteria, no further sampling is planned. - If sampling demonstrates that contaminants are present at concentrations greater than screening levels/criteria, and that contaminant concentrations are greater than upstream conditions (see Phase 1B-GMR to right), further evaluation and/or remedial measures may be warranted.	- If sampling demonstrates conditions adjacent to the Site are less than or equal to those found upstream, no further sampling is planned. - If sampling demonstrates contaminant concentrations are greater than those upstream, and that contaminant concentrations are greater than Action Level criteria (see Phase 1A-GMR to left), further evaluation and/or remediation may be warranted. Further evaluation may consist of an ecological study (i.e., benthic community study; see Phase 2-GMR to the right).	- If the community survey demonstrates that aquatic life in the GMR is not affected by Site-related contaminants, no further sampling is planned. - If the community survey demonstrates that Site-related contaminants impair aquatic life in the GMR and/or the Quarry Pond, further evaluation and/or remedial measures may be warranted.	- If sampling demonstrates that contaminants in sediment are less than screening levels/criteria, no further sampling is planned. - If sampling demonstrates that contaminants are present at concentrations greater than screening levels/criteria, further evaluation and/or remedial measures may be warranted (i.e., acute bioassays on representative Quarry Pond sediments).
iii) Type of problem (decision or estimation)[1]	Decision (Action Level)			

Medium:	Great Miami River (GMR) Sediment			Quarry Pond Sediments
Investigation Phase:	**Phase 1A – GMR**	**Phase 1B – GMR**	**Phase 2 – GMR**	**Phase 1A – Quarry Pond**
Investigation Item:	***Comparison to Human Health and Ecological Screening Values***	***Comparison to Upstream Conditions***	***Benthic Sampling***	***Comparison to Human Health and Ecological Risk Screening Values***
Step 2. Goals of the Study				
iv.a) Decision statement	Determine whether any contaminant concentrations are greater than Industrial Soil RSLs, Residential Soil RSLs, ESLs, or if the sum of Equilibrium Partitioning Sediment Benchmark Toxic Units (∑ESBTUFCV) > 1, or if the organic carbon normalized excess Simultaneously Extracted Metal (∑SEM) > 150 µmol/goc in the GMR sediments near the Site, or if the concentrations of arsenic are greater than its Probable Effects Concentration (PEC).	Determine whether any measurable input of contaminants from the Site, relative to upstream conditions, occurs in the GMR sediments near the Site.	Determine whether any measureable impact to aquatic life in the GMR occurs due to contaminants from the Site, relative to upstream conditions	Determine whether any contaminant concentrations are greater than ESLs, USEPA Industrial soil criteria, USEPA Residential soil criteria, Sum of Equilibrium Partitioning Sediment Benchmark Toxic Units (∑ESBTUFCV) > 1, or organic carbon normalized excess Simultaneously Extracted Metal (∑SEM) > 150 µmol/goc in the on-Site Quarry Pond sediments.
iv.b) Estimation statement & assumptions	--			

Medium:	Great Miami River (GMR) Sediment			Quarry Pond Sediments
Investigation Phase:	**Phase 1A – GMR**	**Phase 1B – GMR**	**Phase 2 – GMR**	**Phase 1A – Quarry Pond**
Investigation Item:	***Comparison to Human Health and Ecological Screening Values***	***Comparison to Upstream Conditions***	***Benthic Sampling***	***Comparison to Human Health and Ecological Risk Screening Values***
Step 3. Identify Information Inputs				
i) Information types needed	Sediment sample analysis is required to assess conditions in the GMR near the Site.		A benthic community survey may be required to assess the impact to aquatic life in the GMR near the Site.	Sediment sample analysis is required to assess conditions in the Quarry Pond.
ii) Information Sources	- New data from the investigation will form the basis of assessment. The results from three previous sediment samples collected from the GMR, as well as results of soil samples will be considered during interpretation of the data obtained. In 1996, Ohio EPA collected sediment samples from the GMR. The sediment samples contained PAHs, PCBs, and metals at concentrations greater than USEPA Residential and/or Industrial Soil RSLs. - Sediment samples will be analyzed for TCL VOCs, TCL SVOCs (including PAHs), TCL pesticides, TCL herbicides, TAL metals, divalent metals (copper, cadmium, mercury, nickel, lead and zinc) using AVS/SEM analyses, and total metals (including arsenic), organic carbon, black carbon, major anions (chloride, fluoride, cyanide, nitrate, nitrite, sulfate, sulfide) and indicator parameters (pH, temperature, conductivity, oxidation reduction potential (ORP), and dissolved oxygen, and reduction-oxidation (REDOX) parameters.		- New data from the community survey will form the basis of assessment. The results from Phase 1A-GMR and 1B-GMR (see left) will be considered during interpretation of the data obtained.	- New data from the investigation will form the basis of assessment. The results from previous sediment samples as well as results of soil samples will be considered during interpretation of the data obtained. In 1996, Ohio EPA collected sediment samples from the Quarry Pond at depths of 15 to 18 ft below the water surface. The sediment samples contained PAHs and arsenic at concentrations greater than USEPA Industrial Soil RSLs. Sediment samples will be analyzed for TCL VOCs, TCL SVOCs (including PAHs), TCL pesticides, TCL herbicides, total TAL metals (including arsenic), divalent metals (copper, cadmium, mercury, nickel, lead and zinc) using AVS/SEM analyses, organic carbon, black carbon, major anions (chloride, fluoride, cyanide, nitrate, nitrite, sulfate, sulfide) and indicator parameters (pH, temperature, conductivity, oxidation reduction potential (ORP), and dissolved oxygen, and reduction-oxidation (REDOX) parameters.

Medium:	Great Miami River (GMR) Sediment			Quarry Pond Sediments
Investigation Phase:	**Phase 1A – GMR**	**Phase 1B – GMR**	**Phase 2 – GMR**	**Phase 1A – Quarry Pond**
Investigation Item:	***Comparison to Human Health and Ecological Screening Values***	***Comparison to Upstream Conditions***	***Benthic Sampling***	***Comparison to Human Health and Ecological Risk Screening Values***
iii) Basis of Action Level	Action levels are: - Industrial Soil RSLs - Final Chronic Values (FCV) for PAHs, ∑ESBTUFCV < 1 - Excess SEM < 150 µmol/goc - PEC values for arsenic - Residential Soil RSLs will be used as an initial screening step to account for early-life susceptibility to mutagens for child receptors	The selected action level is a background threshold value (e.g., 95th percentile) based on upstream conditions.	Population and community level response will be evaluated.	Action levels are: - Industrial Soil RSLs - Final Chronic Values (FCV) for PAHs, ∑ESBTUFCV < 1 - Excess SEM < 150 µmol/goc - PEC values for arsenic - Residential Soil RSLs will be used as an initial screening step to account for early-life susceptibility to mutagens for child receptors
iv) Appropriate sampling & analysis methods	Methods are described in the Field Sampling Plan (CRA, May, 2013), CRA's Standard Operating Procedures, and the Quality Assurance Project Plan (CRA, September 2008). Organic carbon in sediments will be analyzed using the Lloyd Kahn or Walkley-Black methods. PAH results will be evaluated against ∑ESBTUFCV, as detailed in USEPA, 2003. Procedures for the Derivation of Equilibrium Partitioning Sediment Benchmarks (ESBs) for the Protection of Benthic Organisms: PAH Mixtures. EPA-600-R-02-013. Divalent metals results will be evaluated against the organic carbon normalized excess ∑SEM.		A benthic community survey will be completed in accordance with USEPA Rapid Bio assessment Protocols (EPA 841-B-99-002) or OEPA assessment methods (OEPA, 1989. Biological criteria for the protection of aquatic life), depending on the habitat.	Methods are described in the Field Sampling Plan, CRA's Standard Operating Procedures, and the Quality Assurance Project Plan. Organic carbon in sediments will be analyzed using the Lloyd Kahn or Walkley-Black methods. PAH results will be evaluated against ∑ESBTUFCV, as detailed in USEPA, 2003. Procedures for the Derivation of Equilibrium Partitioning Sediment Benchmarks (ESBs) for the Protection of Benthic Organisms: PAH Mixtures. EPA-600-R-02-013. Metals results will be evaluated against the organic carbon normalized excess ∑SEM.

Medium:	Great Miami River (GMR) Sediment			Quarry Pond Sediments
Investigation Phase:	**Phase 1A – GMR**	**Phase 1B – GMR**	**Phase 2 – GMR**	**Phase 1A – Quarry Pond**
Investigation Item:	***Comparison to Human Health and Ecological Screening Values***	***Comparison to Upstream Conditions***	***Benthic Sampling***	***Comparison to Human Health and Ecological Risk Screening Values***
Step 4. Define the Boundaries of the Study				
i) Target population, sample units	The target population are the upper (available) layer of sediments (0 - 6 inches below sediment/water interface), and subsurface sediment (greater than 6 inches below sediment/water interface) in the GMR adjacent to the Site. The sampling units are individual grab samples collected from the near-Site reaches of the GMR. Areas easily accessible to humans and with evidence of use will be targeted for sediment sample locations (e.g., areas where anglers or other recreational users are present; areas where water is approximately 3 ft deep or less and where sediment can support body weight. Depositional areas will also be targeted to identify potential ecological risks. Sediment samples will also be collected in depositional locations immediately downstream of any point discharges identified between the upstream dam and the southern Site boundary. The sediment sample locations may be adjusted based on the location of intermittent drainage pathways (if any).	The target population is the upper (available) layer of sediments (0 - 6 inches below sediment/water interface) and subsurface sediment (greater than 6 inches below sediment/water interface) in the upstream sampling locations. The sampling units are individual grab samples collected from the upstream reaches of the GMR. Areas easily accessible to humans and with evidence of use will be targeted for sediment sample locations (e.g., areas where anglers or other recreational users are present; areas where water is approximately 3 ft deep or less and where sediment can support body weight. Depositional areas will also be targeted to identify potential ecological risks. Sediment samples will be collected in depositional locations immediately downstream of any point discharges identified between the upstream dam and east of the Dryden Road bridge. The sediment sample locations may be adjusted based on the location of intermittent drainage pathways (if any).	The target population is the aquatic life in the GMR in the vicinity of the Site. The sampling units are composite samples collected from the GMR, divided by upstream, near-Site, and downstream reaches. Sampling efforts may be concentrated in near-shore habitats, where most species will be collected.	The target populations are the upper (available) layer of sediments (0 - 6 inches below sediment/water interface), and subsurface sediment (greater than 6 inches below sediment/water interface) in the Quarry Pond. The sampling units are individual grab samples collected from the Quarry Pond. Depositional areas and areas where visual evidence of potential leachate migration is observed will be targeted for sediment sample locations. The sample locations may be adjusted based on the locations of intermittent drainage pathways, storm water runoff pathways, if any are identified, and the results of underwater survey inspections conducted by Ohio EPA, Ohio DNR and the Attorney General's Office – Bureau of Criminal Investigations Office, to include consideration of any areas where foreign objects may have been deposited and the likelihood of sediment contamination may be greater.

Medium:	**Great Miami River (GMR) Sediment**			**Quarry Pond Sediments**
Investigation Phase:	**Phase 1A – GMR**	**Phase 1B – GMR**	**Phase 2 – GMR**	**Phase 1A – Quarry Pond**
Investigation Item:	***Comparison to Human Health and Ecological Screening Values***	***Comparison to Upstream Conditions***	***Benthic Sampling***	***Comparison to Human Health and Ecological Risk Screening Values***
Step 4. Define the Boundaries of the Study				
ii) Specify spatial boundaries	Near-Site sampling locations are those occurring to the west of the Dryden Road bridge (i.e., as surface water passes the Site), and these will be located on the near (south and east) shore of the GMR. Sediment samples will be collected from the top of the sediment layer (i.e., 0 - 6 inches below the sediment/water interface), and subsurface sediments (i.e., greater than 6 inches below the sediment/water interface) in the GMR.	Upstream sampling locations are to the east of the Dryden Road bridge. Sediment samples will be collected from the top of the sediment layer (i.e., 0 - 6 inches below the sediment/water interface), and subsurface sediments (i.e., greater than 6 inches below the sediment/water interface) in the GMR.	Upstream sampling locations are to the east of the Dryden Road bridge. Near-Site sampling locations are those occurring to the west of the Dryden Road bridge (i.e., as surface water passes the Site), and these will be located on the near (south and east) shore of the GMR. Downstream sampling locations are to the south of the City of Dayton Wastewater Treatment Plant.	Sediment samples will be collected from the top of the sediment layer (i.e., 0 - 6 inches below the sediment/water interface), and subsurface sediments (i.e., greater than 6 inches below the sediment/water interface) in the Quarry Pond.
iii) Specify temporal boundaries	The temporal boundaries are indefinite, assuming continued exposure at levels found during sampling. The practical temporal limits are based on exposure assumptions forming the basis for the Action Levels. Initial monitoring will occur over one sampling round. The Respondents will evaluate sediment sample results, and propose additional sampling, if required (e.g., to further evaluate any observed spatial differences, or further define the extent and magnitude of contamination).			The temporal boundaries are indefinite, assuming continued exposure at levels found during sampling. The practical temporal limits are based on exposure assumptions forming the basis for the Action Levels.
iv) Identify any other practical constraints	Sampling may be postponed due to flooding or ice conditions in the GMR. If any dams/weirs are encountered, samples will be collected from the side of the dam closest to the Site (i.e., downstream of any upstream dams, and upstream of any downstream dams).			Sampling may be postponed due to flooding or ice conditions of the Quarry Pond.
v.a) Scale of inference for decision making	Comparisons to Action Levels will be carried out on an individual-location basis.	Comparisons to upstream conditions will be carried out on an individual-location basis.	Criteria in biological indices will be used to evaluate the impacts on aquatic life.	Comparisons to Action Levels will be carried out on an individual-location basis.
v.b) Scale of estimates	--			

Medium:	**Great Miami River (GMR) Sediment**			**Quarry Pond Sediments**
Investigation Phase:	**Phase 1A – GMR**	**Phase 1B – GMR**	**Phase 2 – GMR**	**Phase 1A – Quarry Pond**
Investigation Item:	***Comparison to Human Health and Ecological Screening Values***	***Comparison to Upstream Conditions***	***Benthic Sampling***	***Comparison to Human Health and Ecological Risk Screening Values***
Step 5. Develop the Analytic Approach				
i.a) Specify Action Level	- Residential Soil RSLs will be used as an initial screening step to account for early-life susceptibility to mutagens for child receptors - Industrial Soil RSLs - Final Chronic Values (FCV) for PAHs, ∑ESBTUFCV < 1 - Excess SEM < 150 µmol/goc - PEC values for arsenic	Background Threshold Values based on upstream data, following USEPA's ProUCL Technical Guide (2013)	Criteria in biological indices, consisting of the Index of Well-Being (Gammon 1976; Gammon *et al. 1981)*, the Index of Biotic Integrity (Karr 1981; Fausch *et al.* 1984), and the Invertebrate Community Index (DeShon *et al.* unpublished)	- Residential Soil RSLs will be used as an initial screening step to account for early-life susceptibility to mutagens for child receptors - Industrial Soil RSLs - Final Chronic Values (FCV) for PAHs, ∑ESBTUFCV < 1 - Excess SEM < 150 µmol/goc - PEC values for arsenic
i.b) Specify estimator	--	--	--	--
ii.a) Specify population parameter of interest and theoretical decision rule	Individual observations at near-Site sampling locations.		Cumulative observations at near-Site sampling locations.	Individual observations at near-Site sampling locations.
ii.b) Specify estimation procedure	--	--	--	--

Medium:	**Great Miami River (GMR) Sediment**			**Quarry Pond Sediments**
Investigation Phase:	**Phase 1A – GMR**	**Phase 1B – GMR**	**Phase 2 – GMR**	**Phase 1A – Quarry Pond**
Investigation Item:	***Comparison to Human Health and Ecological Screening Values***	***Comparison to Upstream Conditions***	***Benthic Sampling***	***Comparison to Human Health and Ecological Risk Screening Values***
Step 6. Specify Performance or Acceptance Criteria				
i.a) Set baseline (null) and alternative hypotheses	Baseline H_0: sediment concentrations are less than Action Levels Alternative H_1: sediment contaminant concentrations are greater than Action Levels	Baseline H_0: Concentrations of Site-related chemicals in near-Site sediments are no different than upstream Alternative H_1: Concentrations of Site-related chemicals in near-Site sediments contain contaminants at concentrations greater than upstream conditions	Baseline H_0: aquatic ecosystem in near-Site reaches are no different than upstream Alternative H_1: aquatic ecosystem in near-Site reaches is impaired in comparison to upstream conditions.	Baseline H_0: sediment concentrations are less than Action Levels Alternative H_1: sediment contaminant concentrations are greater than Action Levels
i.b) Specify how uncertainty accounted for in estimate	--	--	--	--
ii.a) Determine impact of decision errors (false positives/negatives)	N/A: no statistical test is employed (direct comparison to Action Levels)	- If a false positive (Type I) error occurs, unnecessary additional investigation may occur. - If a false negative (Type II) error occurs, conditions that are not due to background concentrations and pose potential risk to aquatic ecosystem and/or human receptors could persist.	- If a false positive (Type I) error occurs, unnecessary additional investigation may occur. - If a false negative (Type II) error occurs, conditions posing potential risk to the aquatic ecosystem could persist.	N/A: no statistical test is employed (direct comparison to Action Levels)
ii.b) Specify confidence level for estimate	--	--	--	--

Medium:	Great Miami River (GMR) Sediment			Quarry Pond Sediments
Investigation Phase:	**Phase 1A – GMR**	**Phase 1B – GMR**	**Phase 2 – GMR**	**Phase 1A – Quarry Pond**
Investigation Item:	***Comparison to Human Health and Ecological Screening Values***	***Comparison to Upstream Conditions***	***Benthic Sampling***	***Comparison to Human Health and Ecological Risk Screening Values***
Step 6. Specify Performance or Acceptance Criteria				
iii) Specify "gray region" for test	N/A: no statistical test is employed (direct comparison to Action Levels)	For comparisons to upgradient conditions, the gray region will be set equal to a difference in means (on-Site and upgradient) of one standard deviation of the upgradient data.	--	N/A: no statistical test is employed (direct comparison to Action Levels)
iv.a) Set tolerable limits on decision errors	N/A: no statistical test is employed (direct comparison to Action Levels)	- The Background Threshold Values will be calculated using a 95 percent confidence level, making the false positive rate no greater than 5 percent. - Limits on the false negative rate are not appropriate for comparisons of individual results to threshold values.	--	N/A: no statistical test is employed (direct comparison to Action Levels)
iv.b) Specify performance or acceptance criteria	Total sediment concentrations will be used in the comparison to Action Levels, rather than subtracting background concentrations, for evaluation in the Ecological Risk Assessment.		--	Total sediment concentrations will be used in the comparison to Action Levels, rather than subtracting background concentrations, for evaluation in the Ecological Risk Assessment.

Medium:	Great Miami River (GMR) Sediment			Quarry Pond Sediments
Investigation Phase:	**Phase 1A – GMR**	**Phase 1B – GMR**	**Phase 2 – GMR**	**Phase 1A – Quarry Pond**
Investigation Item:	***Comparison to Human Health and Ecological Screening Values***	***Comparison to Upstream Conditions***	***Benthic Sampling***	***Comparison to Human Health and Ecological Risk Screening Values***
Step 7. Develop the Plan for Obtaining Data				
i) Select sampling design	Near-Site samples will be collected in one sampling event close to the proximate (south/east) shore of the GMR at (i) the upstream edge of the Site, including both a near-shore and far-shore sample; (ii) mid-Site, downgradient of monitoring wells containing highest VOC concentrations on the side of the Site nearest the river; (iii) further downstream in the mid-Site region, halfway between (ii) and (iv); (iv) downstream of the main Site, upstream of the City's WWTP outlet; and (v) downstream of the entire Site. Samples will be biased towards locations with fine-grained sediments with higher organic carbon (based on visual observation). Proposed sample locations will be adjusted in the field to ensure that the samples are collected from sediments most representative of potential worst-case issues.	Upstream samples will be collected in one sampling event at 9 locations to provide a suitable data set (per USEPA's ProUCL Technical Guide, 2010) for the calculation of Background Threshold Values. Upstream samples will be collected along 3 transects of 3 samples each, regularly spaced downstream of the upstream dam, and upstream low-head of the Site. Near-Site samples will be collected as described in Phase 1A (see left).	Near-Site samples will be collected close to the proximate (south/east) shore of the GMR at (i) the upstream edge of the Site, including both a near-shore and far-shore sample; (ii) mid-Site, downgradient of monitoring wells containing highest VOC concentrations on the side of the Site nearest the river; (iii) further downstream in the mid-Site region, halfway between (ii) and (iv); (iv) downstream of the main Site, upstream of the City's WWTP outlet; and (v) downstream of the entire Site. The sampling effort may be concentrated in near-shore habitats where most species will be collected and will be biased toward areas where the greatest sediment impacts were identified during the Phase 1A and 1B investigations.	Up to 9 samples will be collected from the Quarry Pond, along 3 transects of 3 samples each. Samples will be biased towards locations with fine-grained sediments with higher organic carbon (based on visual observation). Proposed sample locations will be adjusted in the field to ensure that the samples are collected from sediments most representative of potential worst-case issues.

Medium:	**Great Miami River (GMR) Sediment**			**Quarry Pond Sediments**
Investigation Phase:	**Phase 1A – GMR**	**Phase 1B – GMR**	**Phase 2 – GMR**	**Phase 1A – Quarry Pond**
Investigation Item:	***Comparison to Human Health and Ecological Screening Values***	***Comparison to Upstream Conditions***	***Benthic Sampling***	***Comparison to Human Health and Ecological Risk Screening Values***
Step 7. Develop the Plan for Obtaining Data				
ii) Specify/evaluate key assumptions supporting the design	The mechanisms of contaminant transport from the Site to river sediments, i.e., via groundwater migration and seepage or via erosion and runoff, would result in greatest impacts (if any) near-shore and potentially, due to groundwater seepage, midstream. Sampling locations have been selected reflecting this, and covering different potential directions of transport and deposition, covering the full range of possibilities from the Site.	The calculation Background Threshold Values (statistical limits on an upper percentile, e.g., 95th) for the upstream population of sediments depends on data characteristics (e.g., distribution and proportion of detected values), as fully discussed in the USEPA ProUCL Technical Guide (2013). Additionally, the presence of outlying values will be tested, and if present their impact on the values obtained evaluated.	The mechanisms of contaminant transport from the Site to river sediments, i.e., via groundwater migration and seepage or via erosion and runoff, would result in greatest impacts (if any) near-shore. Sampling locations have been selected reflecting this, and covering different potential directions of transport and deposition, covering the full range of possibilities from the Site.	The mechanisms of contaminant transport from the Site to pond sediments, i.e., via groundwater migration and seepage or via erosion and runoff, would result in greatest impacts (if any) near-shore. Sampling locations have been selected reflecting this, and covering different potential directions of transport and deposition, covering the full range of possibilities from the Site.

Notes:

[1] If investigating a "decision problem", follow items ending in ".a" in subsequent DQO steps (e.g., "ii.a" or "iii.a").

If investigating an "estimation problem", follow ".b" items.

Once the baseline risk assessment for OU2 has been performed, possible remedial goals (PRGs) will be derived from the calculator using site-specific risks.

-- Item not applicable for the type of problem (decision vs. estimation) investigated.

The planning team includes:
Respondents: Ken Brown (ITW); Jim Campbell (ITW); Bryan Heath (NCR); Wendell Barner (KELSEY HAYES CO.)
Steve Quigley (CRA project manager);

Wesley Dyck, Daniela Araujo (CRA statistics expert);

April Gowing, Steve Harris, Vincent Nero and Dan Smith (CRA risk assessment experts);

Rawa Fleisher, Angela Bown (CRA chemists/quality assurance staff);

Julian Hayward, Valerie Chan and Adam Loney (CRA project engineers); Alan Deal (CRA project hydro-geologist);

Leslie Patterson (USEPA Regional Project Manager); Madelyn Smith (Ohio EPA representative); and property owner stakeholders.

11-6 - Flood Plain

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Site-Specific Risk Values***	***Comparison to Background Reference Conditions***	***Additional Sampling (if necessary) to Develop Risk Assessment Exposure Estimates***
Step 1. State the Problem			
i) Problem description	Potential risk to industrial workers from exposure to on-Site soils has been identified in a human health risk assessment. It is not known if potential soil contamination in the floodplain (a) poses risks to human receptors due to recreational use, and (b) is a result of migration from the Site. Analysis of floodplain soil samples is required to make these assessments. It is also unknown whether floodplain soils pose ecological risks either in-situ or if soils are eroded and enter the Great Miami River (GMR).		If, during Phase 1, floodplain soil containing Site-related contaminants at concentrations greater than screening values and background reference conditions is identified, characterization of conditions within the exposure unit (i.e., nature and extent of contamination) is required for risk assessment purposes.
ii) Planning team	See note at bottom		
iii) Conceptual model	- Cover material at the Site is limited or non-existent, which could lead to erosional run-off of contaminants towards the floodplain of the GMR, which may pose a risk to human and ecological receptors. - In addition, movement of contaminants in dust particles carried by wind may result in deposition of contaminants off-Site. - Soil contaminants are assumed to have been deposited by erosion and mixed by subsequent flooding events. -The floodplain can serve as habitat for small mammals and birds.		
iv) General intended use for data	The data collected will be screened against health-based and ecological risk values. The goal of the investigation is to identify human health direct contact, ingestion, and inhalation risks, and ecological risks associated with surficial soil in the floodplain and determine the magnitude and extent of contamination from Site-related contaminants.	The data collected from sampling locations along the Site's boundaries will be compared to upstream floodplain soil conditions, to determine if there are any measurable inputs of contaminants from the Site and determine the magnitude and extent of contamination from Site-related contaminants. The data collected will ultimately be used in the Baseline Risk Assessment for OU2.	The collected data will be used to determine the magnitude and extent of contamination from Site-related contaminants, and generate human health and/or ecological exposure estimates for a risk assessment. The data collected will ultimately be used in the Baseline Risk Assessment for OU2.
v) Resources, constraints, deadlines	Sufficient resources will be committed to sample off-Site soil under the OU2 RI/FS work plan. Sampling may be postponed due to flooding, and could be constrained due to access agreements in off-Site areas.		

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Site-Specific Risk Values***	***Comparison to Background Reference Conditions***	***Additional Sampling (if necessary) to Develop Risk Assessment Exposure Estimates***
Step 2. Goals of the Study			
i) Primary study question	Do near-Site floodplain soils contain Site-related contaminants at concentrations that pose a potential risk to receptors, based on the use of screening criteria, i.e., Residential Soil RSLs, and/or Site-specific risk-based values? If so, what are the risks?	Does the Site add contaminants to soil in the floodplain of the GMR near the Site?	What are the risks from floodplain soils contaminated by Site-related sources?
ii) Alternate outcomes or actions	- If sampling demonstrates that contaminants in soil are less than risk-based screening levels/criteria, no further sampling is planned. - If sampling demonstrates that contaminant concentrations are greater than screening levels/criteria, and greater than background reference conditions (see Phase 1B to right), further evaluation and/or remedial measures may be warranted.	- If sampling demonstrates conditions adjacent to the Site are not greater than those found in background reference soils, no further sampling is planned. - If sampling demonstrates conditions are greater than background, and that contaminant concentrations are greater than Action Level criteria (see Phase 1A to left), further evaluation and/or remediation may be warranted.	- If sampling demonstrates that health risks are acceptable, no further action is required. - If sampling demonstrates unacceptable risks, further evaluation, risk management and/or remediation would be required.
iii) Type of problem (decision or estimation)[1]	Decision (Action Level)	Decision (Action Level)	Estimation
iv.a) Decision statement	Determine whether any contaminant concentrations are greater than USEPA Residential Soil RSLs or Site-specific risk values in off-Site floodplain soil near the Site.	Determine whether any measurable input of contaminants from the Site, relative to background reference conditions, occurs in near-Site floodplain soil near the Site.	--
iv.b) Estimation statement & assumptions	--	--	The parameter of interest is 95% UCL of the mean (for estimating inhalation, dermal exposure, and ingestion risks, etc.) of soil contaminant concentrations within an identified off-Site exposure area. Section 5.2 of the OU2 RI/FS Work Plan details the proposed exposure units.

Investigation Phase:	**Phase 1A**	**Phase 1B**	**Phase 2**
Investigation Item:	***Comparison to Site-Specific Risk Values***	***Comparison to Background Reference Conditions***	***Additional Sampling (if necessary) to Develop Risk Assessment Exposure Estimates***
Step 3. Identify Information Inputs			
i) Information types needed	- Soil sample analysis is required to assess conditions in the floodplain of the GMR near the Site. - Soil samples will be collected at locations adjacent to (i.e., downgradient of) known on-Site issues, and also biased toward erosional areas. -Background soil contaminant concentrations (from Worksheet 11-1)		- This would be a supplemental data collection effort, with analyses performed on soil samples obtained to fill in any data gaps across the exposure area.
ii) Information Sources	- New data from the investigation will form the basis of assessment. The results from three previous sediment samples collected from the GMR will be considered during interpretation of the data obtained.		- New data from the investigation will form the basis of assessment. Available previous validated data (e.g., from Phase 1), within the exposure area would also be used.
iii) Basis of Action Level	Action Levels are: - USEPA Residential Soil RSLs -USEPA RCRA ESLs	The selected Action Level is a Background Threshold Value (e.g., 95th percentile) based on background reference conditions.	--
iv) Appropriate sampling & analysis methods	Methods are described in the Field Sampling Plan (CRA, May, 2013) and the Quality Assurance Project Plan (CRA, September 2008).		

[1] Ecological Screening Levels (ESLs) will be presented and defined in the Screening Level Environmental Risk Assessment (SLERA) work plan

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Site-Specific Risk Values***	***Comparison to Background Reference Conditions***	***Additional Sampling (if necessary) to Develop Risk Assessment Exposure Estimates***
Step 4. Define the Boundaries of the Study			
i) Target population, sample units	The target population is surficial soil on the floodplain of the GMR near the Site. CRA has defined the single exposure unit of the floodplain to be the length of bike path/recreational trail adjacent to the Site boundary, as this is the area of heaviest traffic in which receptors are most likely exposed to any Site-related contamination, as discussed in Section 5.2 of the OU2 RI/FS Work Plan. The sampling units are individual samples collected from surface soil located between the Site embankment and the bike path.	The sampling units are individual samples collected from surface soil from background reference sampling locations. Background reference sampling locations will be identified in areas outside a reasonable zone of potential influence (via surface runoff or substantial airborne dust deposition) for the Site.	Target population is surficial floodplain soils comprising the exposure unit for assessment of exposure risks for human receptors.
ii) Specify spatial boundaries	The spatial boundaries of the floodplain soil sampling locations are the floodplain soil of the GMR, located between the Site embankment and the bike path/recreational trail.	The spatial boundaries of the floodplain soil sampling locations are the floodplain soil of the GMR, located between the Site embankment and the bike path/recreational trail.	The spatial boundaries are the limits of the surficial soils in the identified off-Site exposure area (based on Phase 1 findings).
iii) Specify temporal boundaries	The temporal boundaries are indefinite, assuming continued exposure at levels found during sampling. The practical temporal limits are based on the exposure assumptions of the Action Levels.		
iv) Identify any other practical constraints	Due to the presence of a high pressure gas line in the floodplain, soil samples will be hand-dug. If different surficial soil substrates are encountered (e.g., silt vs. sand vs. clay), these differences may require additional sampling (e.g., further reference samples) to appropriately evaluate potential Site-related impacts. Off-Site sampling may be restricted by permission of property owners, e.g. for background locations.		Further practical constraints are not anticipated for sampling of floodplain soils near to the Site.
v.a) Scale of inference for decision making	Comparisons to Action Levels will be carried out on an individual-location basis.	Comparisons to background reference conditions will be carried out on an individual-location basis.	--
v.b) Scale of estimates	--	--	The scale of the exposure estimate is to be identified in a Site-specific risk assessment.

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Site-Specific Risk Values***	***Comparison to Background Reference Conditions***	***Additional Sampling (if necessary) to Develop Risk Assessment Exposure Estimates***
Step 5. Develop the Analytic Approach			
i.a) Specify Action Level	1) USEPA Residential Soil RSLs 2) USEPA RCRA ESLs	Background Threshold Values based on background reference data, following USEPA's ProUCL Technical Guide (2013)	--
i.b) Specify estimator	--	--	The arithmetic mean (per USEPA RAGS requirements) surface soil concentration of each contaminant that is greater than screening criteria.
ii.a) Specify population parameter of interest and theoretical decision rule	Individual observations at near-Site sampling locations.		--
ii.b) Specify estimation procedure	--	--	The study will estimate the mean concentration of the exposure unit population represented by the soil samples obtained.

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Site-Specific Risk Values***	***Comparison to Background Reference Conditions***	***Additional Sampling (if necessary) to Develop Risk Assessment Exposure Estimates***
Step 6. Specify Performance or Acceptance Criteria			
i.a) Set baseline (null) and alternative hypotheses	Baseline H_0: soil sample concentrations are less than Action Levels Alternative H_1: soil samples contaminated at concentrations greater than Action Levels	Baseline H_0: near-Site floodplain soil sample concentrations are no different than reference Alternative H_1: near-Site floodplain soil samples contain contaminants at concentrations greater than reference conditions	--
i.b) Specify how uncertainty accounted for in estimate	--	--	Uncertainty will be accounted for using a confidence interval on the population mean (per USEPA RAGS guidance).
ii.a) Determine impact of decision errors (false positives/negatives)	N/A: no statistical test is employed (direct comparison to Action Levels)	- If a false positive (Type I) error occurs, unnecessary additional investigation (Phase 2) may occur. - If a false negative (Type II) error occurs, conditions that are not due to background concentrations of contaminants and that pose potential health risks to receptors persist.	--
ii.b) Specify confidence level for estimate	--	--	The confidence level of the estimate will be 95 percent, unless specified otherwise (based on data distribution and/or the presence of non-detect results) in USEPA's ProUCL Technical Guide (2013).
iii) Specify "gray region" for test	N/A: no statistical test is employed (direct comparison to Action Levels)	N/A: since comparing individual concentrations against reference conditions, no statistical test is employed	--
iv.a) Set tolerable limits on decision errors	N/A: no statistical test is employed (direct comparison to Action Levels)	The Background Threshold Values will be calculated using a 95 percent confidence level, making the false positive rate no greater than 5 percent. Limits on the false negative rate are not appropriate for comparisons of individual results to threshold values.	--

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Site-Specific Risk Values***	***Comparison to Background Reference Conditions***	***Additional Sampling (if necessary) to Develop Risk Assessment Exposure Estimates***
Step 6. Specify Performance or Acceptance Criteria			
iv.b) Specify performance or acceptance criteria	--	--	The lesser value of the 95 percent UCL on the population mean or the maximum individual measurement will be used for comparison to risk-based criteria.

Investigation Phase:	Phase 1A	Phase 1B	Phase 2
Investigation Item:	***Comparison to Site-Specific Risk Values***	***Comparison to Background Reference Conditions***	***Additional Sampling (if necessary) to Develop Risk Assessment Exposure Estimates***
Step 7. Develop the Plan for Obtaining Data			
i) Select sampling design	Near-Site surficial soil samples will be collected on the floodplain. These include (i) the upgradient edge of the Site; (ii) mid-Site, downgradient of monitoring wells containing highest VOC concentrations on the side of the Site nearest the river; (iii) further downgradient, halfway between (ii) and (iv); and (iv) at the furthest downgradient boundary of the Site. Approximately 15 surficial soil samples will be collected from the near-Site portion of the floodplain around the recreational trail.	Background reference samples will be collected at 10 locations to provide a suitable data set (per USEPA's ProUCL Technical Guide, 2013) for the calculation of Background Threshold Values. Near-Site samples will be collected as described in Phase 1A (see left).	A minimum of 10 samples, per USEPA's ProUCL Technical Guide (2013), spaced on a regular grid with random origin (i.e., a systematic random sampling design), will be obtained for each exposure area identified in the risk assessment. Samples collected during Phase 1 will be included within the 10 sample data set.
ii) Specify/evaluate key assumptions supporting the design	Contaminant transport from the Site to floodplain soils via erosion/runoff is expected to result in greatest impacts (if any) closest to the Site at the base of the embankment. Sampling locations have been selected reflecting this (i.e., including locations biased towards areas with highest contamination potential), and cover all different potential directions of transport/deposition from the Site.	The calculation Background Threshold Values (statistical limits on an upper percentile, e.g. 95th) for the reference population of surficial soils depends on data characteristics (e.g., distribution and proportion of detected values), as fully discussed in the USEPA ProUCL Technical Guide (2013). Additionally, the presence of outlying values will be tested, and if present their impact on the values obtained evaluated.	The calculation of 95 percent upper confidence limits on a population mean makes assumptions of data characteristics (e.g., distribution and proportion of detected values), as fully discussed in the USEPA ProUCL Technical Guide (2013). Additionally, the presence of outlying values will be tested, and if present their impact on the values obtained evaluated.

Notes:

[1] If investigating a "decision problem", follow items ending in ".a" in subsequent DQO steps (e.g., "ii.a" or "iii.a").

If investigating an "estimation problem", follow ".b" items.

Once the baseline risk assessment for OU2 has been performed, possible remedial goals (PRGs) will be derived from the calculator using site-specific risks.

-- Item not applicable for the type of problem (decision vs. estimation) investigated.

The planning team includes:
Respondents: Ken Brown (ITW); Jim Campbell (ITW); Bryan Heath (NCR); Wendell Barner (KELSEY HAYES CO.)
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Julian Hayward, Valerie Chan and Adam Loney (CRA project engineers); Alan Deal (CRA project hydrogeologist);

Leslie Patterson (USEPA Regional Project Manager); Madelyn Smith (Ohio EPA representative); and property owner stakeholders.

QAPP Worksheet #12 - Measurement Performance Criteria

12-1 - VOCs

Matrix: Water and Solid
Analytical Group or Method: VOA / SW846 8260B
Concentration Level: Low

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field Duplicates	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 30%
Analytical Accuracy / Bias (laboratory)	Laboratory Control Samples	Analyte-specific (see Appendix D-1, SOP NC-MS-019, Table 7)
Analytical Accuracy / Bias (matrix interference)	Matrix Spike Duplicates	Analyte-specific (see Appendix D-1, SOP NC-MS-019, Table 7)
Overall accuracy / bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ RL
Completeness	See Worksheet #34	See Worksheet #34

12-2 - SVOCs

Matrix: Water and Solid
Analytical Group or Method: SVOC / SW846 8270
Concentration Level: Standard

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field Duplicates	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 30%
Analytical Accuracy / Bias (laboratory)	Laboratory Control Samples	Analyte-specific (see Appendix D-2, SOP NC-MS-018, Table 6)
Analytical Accuracy / Bias (matrix interference)	Matrix Spike Duplicates	Analyte-specific (see Appendix D-2, SOP NC-MS-018, Table 7)
Overall accuracy / bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ RL
Completeness	See Worksheet #34	See Worksheet #34

12-3 - PCBs

Matrix: Water and Solid
Analytical Group or Method: PCB / SW846 8082
Concentration Level: All

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field / Sample Duplicates	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.
Analytical Accuracy / Bias (laboratory)	Laboratory Control Samples	Analyte-specific (see Appendix D-3, SOP NC-GC-038, Table C4)
Analytical Accuracy / Bias (matrix interference)	Matrix Spike Duplicates	Analyte-specific (see Appendix D-3, SOP NC-GC-038, Table C4)
Overall accuracy / bias (contamination)	Equipment/Method Blanks	No target analyte concentrations ≥ 1/2 LOQ
Completeness	See Worksheet #34	See Worksheet #34

12-4 - Pesticides

Matrix: Water and Solid
Analytical Group or Method: Pesticides / SW846 8081A
Concentration Level: All

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field / Sample Duplicates	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.
Analytical Accuracy / Bias (laboratory)	Laboratory Control Samples	Analyte-specific (see Appendix D-3, SOP NC-GC-038, Table B5 & Table B6)
Analytical Accuracy / Bias (matrix interference)	Matrix Spike Duplicates	Analyte-specific (see Appendix D-3, SOP NC-GC-038, Table B5 & Table B6)
Overall accuracy / bias (contamination)	Equipment/Method Blanks	No target analyte concentrations ≥ RL
Completeness	See Worksheet #34	See Worksheet #34

12-5 - Herbicides

Matrix: Water and Solid
Analytical Group or Method: Herbicides / SW846 8151A
Concentration Level: All

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field / sample Duplicates	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.
Analytical Accuracy / Bias (laboratory)	Laboratory Control Samples	Analyte-specific (see Appendix D-3, SOP NC-GC-038, Table D3)
Analytical Accuracy / Bias (matrix interference)	Matrix Spike Duplicates	Analyte-specific (see Appendix D-3, SOP NC-GC-038, Table D3)
Overall accuracy / bias (contamination)	Equipment / Method Blanks	No target analyte concentrations ≥ RL
Completeness	See Worksheet #34	See Worksheet #34

12-6 - Dissolved Gases

Matrix: Water
Analytical Group or Method: GC RSK-175
Concentration Level: N/A

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field / sample Duplicates	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL.
Analytical Accuracy / Bias (laboratory)	Laboratory Control Samples	Analyte-specific (see Appendix D-4, SOP NC-GC-032)
Analytical Accuracy / Bias (matrix interference)	Matrix Spike Duplicates	Analyte-specific (see Appendix D-4, SOP NC-GC-032)
Overall accuracy / bias (contamination)	Equipment / Method Blanks	No target analyte concentrations ≥ RL
Completeness	See Worksheet #34	See Worksheet #34

12-7 - TAL Metals and Inorganics

Matrix: Water and Solid
Analytical Group or Method: SW846 6010 and EPA 200.7
Concentration Level: All

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field / Sample Duplicates	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.
Analytical Accuracy / Bias (laboratory)	Laboratory Control Samples	Analyte-specific (see Appendix D-5, SOP NC-MT-012, Table II)
Analytical Accuracy / Bias (matrix interference)	Matrix Spike Duplicates	Analyte-specific (see Appendix D-5, SOP NC-MT-012, Table II)
Overall accuracy / bias (contamination)	Equipment / Method Blanks	No target analyte concentrations ≥ RL
Completeness	See Worksheet #34	See Worksheet #34

12-8 - Mercury

Matrix: Water and Solid
Analytical Group or Method: Mercury / MCAWW 245.1; SW846 7470A, 7471A, 7471B
Concentration Level: All

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field / Sample Duplicates	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.
Analytical Accuracy / Bias (laboratory)	Laboratory Control Samples	Analyte-specific (see Appendix D-6, SOP NC-MT-014, Table 1)
Analytical Accuracy / Bias (matrix interference)	Matrix Spike Duplicates	Analyte-specific (see Appendix D-6, SOP NC-MT-014, Table 1)
Overall accuracy / bias (contamination)	Equipment / Method Blanks	No target analyte concentrations ≥ RL
Completeness	See Worksheet #34	See Worksheet #34

12-9 - Cyanide

Matrix: Water and Solid
Analytical Group or Method: Cyanide / SW846 9012A, 9012B; EPA 335.1, 335.2, 335.2 (CLP-M), 335.4; SM 4500-CN-E, 4500-CN-G
Concentration Level: All, total

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field / Sample Duplicates	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.
Analytical Accuracy / Bias (laboratory)	Laboratory Control Samples	Analyte-specific (see Appendix D-7, SOP NC-WC-031)
Analytical Accuracy / Bias (matrix interference)	Matrix Spike Duplicates	Analyte-specific (see Appendix D-7, SOP NC-WC-031)
Overall accuracy / bias (contamination)	Equipment / Method Blanks	No target analyte concentrations ≥ 1/2 LOQ
Completeness	See Worksheet #34	See Worksheet #34

12-10 -Total Solids, Percent Moisture, and Total Settable Solids

Matrix: Solid
Analytical Group or Method: Percent Moisture-Total Solids / EPA 160.3 Modified, 160.5; ASTM D2216-98
Concentration Level: N/A

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field / Sample Duplicates	Solid: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.

12-11 - Alkalinity

Matrix: Water
Analytical Group or Method: Alkalinity / SM2320B and EPA 310.1
Concentration Level: All

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field / Sample Duplicates	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL.
Analytical Accuracy / Bias (laboratory)	Laboratory Control Samples	Analyte-specific (see Appendix D-9, SOP NC-WC-093)
Analytical Accuracy / Bias (matrix interference)	Matrix Spike Duplicates	Analyte-specific (see Appendix D-9, SOP NC-WC-093)
Overall accuracy / bias (contamination)	Equipment / Method Blanks	No target analyte concentrations ≥ RL
Completeness	See Worksheet #34	See Worksheet #34

12-12 - Anions

Matrix: Water
Analytical Group or Method: Anions / SW846 9056A; EPA 300.0A
Concentration Level: All

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field / Sample Duplicates	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL.
Analytical Accuracy / Bias (laboratory)	Laboratory Control Samples	Analyte-specific (see Appendix D-10, SOP NC-WC-084) 90-110% recovery
Analytical Accuracy / Bias (matrix interference)	Matrix Spike Duplicates	Analyte-specific (see Appendix D-10, SOP NC-WC-084) 80-120% recovery and >20% RPD
Overall accuracy / bias (contamination)	Equipment / Method Blanks	No target analyte concentrations ≥ RL
Completeness	See Worksheet #34	See Worksheet #34

12-13 - Organic Carbon (Dissolved)

Matrix: Water and Soil
Analytical Group or Method: TOC-DOC / EPA 415.1; SW846 9060, 9060A; SM5310C; Walkley Black
Concentration Level: All

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field / Sample Duplicates	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.
Analytical Accuracy / Bias (laboratory)	Laboratory Control Samples	Analyte-specific for water matrices (see Appendix D-11, SOP NC-WC-017) Analyte-specific for non-water matrices (see Appendix D-18, SOP NC-WC-018
Analytical Accuracy / Bias (matrix interference)	Matrix Spike Duplicates	Analyte-specific for water matrices (see Appendix D-11, SOP NC-WC-017) Analyte-specific for non-water matrices (see Appendix D-18, SOP NC-WC-018
Overall accuracy / bias (contamination)	Equipment / Method Blanks	No target analyte concentrations ≥ RL
Completeness	See Worksheet #34	See Worksheet #34

12-14 - pH

Matrix: Water and Solid
Analytical Group or Method: SW846 9040B, 9040C, 9041A, 9045C, 9045D; EPA 150.1; SM 4500H*B
Concentration Level: N/A

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field / Sample Duplicates	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.
Analytical Accuracy / Bias (laboratory)	Laboratory Control Samples	Analyte-specific (see Appendix D-12, SOP NC-WC-010)
Completeness	See Worksheet #34	See Worksheet #34

12-15 - Sulfide

Matrix: Water and Solid
Analytical Group or Method: SW846 9030B, 9034; EPA 376.1; SM 4500-S2-F
Concentration Level: All

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field / Sample Duplicates	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.
Analytical Accuracy / Bias (laboratory)	Laboratory Control Samples	Analyte-specific (see Appendix D-13, SOP NC-WC-060)
Analytical Accuracy / Bias (matrix interference)	Matrix Spike Duplicates	Analyte-specific (see Appendix D-13, SOP NC-WC-060)
Overall Accuracy / bias (contamination)	Equipment / Method Blanks	No target analyte concentrations ≥ RL
Completeness	See Worksheet #34	See Worksheet #34

12-16 - Hardness

Matrix: Water
Analytical Group or Method: General Chemistry / SM 2340C and EPA 130.2
Concentration Level: All

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field Duplicates	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL.
Analytical Accuracy / Bias (laboratory)	Laboratory Control Samples	Analyte-specific (see Appendix D-14, SOP NC-WC-036)
Analytical Accuracy / Bias (matrix interference)	Matrix Spike Duplicates	Analyte-specific (see Appendix D-14, SOP NC-WC-036)
Overall accuracy / bias (contamination)	Method Blanks	No target analyte concentrations ≥ RL
Completeness	See Worksheet #34	See Worksheet #34

12-17 - Flashpoint

Matrix: Water and Solid
Analytical Group or Method: SW846 1010 Closed Cup Flashpoint
Concentration Level: N/A

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Overall Precision	Field Duplicates	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.
Analytical Accuracy / Bias (laboratory)	Laboratory Control Samples	Analyte-specific (see Appendix D-15, SOP NC-WC-034)
Completeness	See Worksheet #34	See Worksheet #34

12-18 - Dioxins and Furans

Matrix: Water and Solid
Analytical Group or Method: Dioxin / Furans SW846 8290A, TO-9A
Concentration Level: All

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Precision	Field Duplicates	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.
Analytical Accuracy / Bias (laboratory)	Laboratory Control Samples	Analyte-specific (See list #1 Below)
Analytical Accuracy / Bias (matrix interference)	Labeled Internal Standards (Spiked Pre-Extraction)	40 to 135% Recovery
Contamination	Method Blanks	No target analyte concentrations ≥ RL
Completeness	See Worksheet #34	See Worksheet #34

List #1 TestAmerica Knoxville 8290A Laboratory Control Sample Acceptance Criteria[1]

	SOIL		WATER	
Compound	**LCS Lower Control Limit**	**LCS Upper Control Limit**	**LCS Lower Control Limit**	**LCS Upper Control Limit**
2,3,7,8-TCDD	79	129	77	127
1,2,3,7,8-PeCDD	79	129	78	128
1,2,3,4,7,8-HxCDD	73	123	73	123
1,2,3,6,7,8-HxCDD	74	124	72	127
1,2,3,7,8,9-HxCDD	70	124	76	126
1,2,3,4,6,7,8-HpCDD	73	123	73	123
OCDD	75	125	75	125
2,3,7,8-TCDF	75	125	74	124
1,2,3,7,8-PeCDF	74	124	74	124
2,3,4,7,8-PeCDF	75	125	74	124
1,2,3,4,7,8-HxCDF	75	125	75	125
1,2,3,6,7,8-HxCDF	76	126	75	125
2,3,4,6,7,8-HxCDF	76	126	76	126
1,2,3,7,8,9-HxCDF	77	127	76	126
1,2,3,4,6,7,8-HpCDF	77	127	71	121
1,2,3,4,7,8,9-HpCDF	73	123	73	123
OCDF	49	128	68	132

LCS acceptance criteria are based on historical data and are subject to update1[1]

12-19 - VOCs in Air

Matrix: Air
Analytical Group or Method: Volatile Organics / TO-15
Concentration Level: All

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
Contamination	Method Blank	No Target Compound >=RL
Accuracy / Bias	Laboratory Control Sample	70-130% recovery with provisory analytes within 60-140%. Marginal exceedence limit of 60-140% / 50-150% allowed based on # of target analytes
Accuracy / Bias	Surrogate	60-140% R
Precision	Laboratory Duplicate	Advisory limit of RPD ≤ 50% for air samples

QAPP Worksheet #13 - Secondary Data Criteria and Limitations

DATA TYPE	*SOURCE*	*DATA USES RELATIVE TO THE CURRENT PROJECT*	*FACTORS AFFECTING THE RELIABILITY OF DATA AND LIMITATIONS ON DATA USE*
Interviews and Aerial Inspection	Ohio EPA, Preliminary Assessment for the South Dayton Dump and Landfill (1985)	• Preliminary identification of hazardous chemicals at the Site that pose a potential threat to groundwater and the GMR. • Identified groundwater flow direction.	• Based on CRA's review, it appears that the determination of groundwater flow directions was not made on the basis of monitoring well information — no wells were present at the time
Screening Site Inspection (SSI) including collection and analysis of surface and subsurface soil samples	Ecology and Environment, Inc. (EEI), Screening Site Inspection Report for South Dayton Dump, Moraine, Ohio. Prepared by EEI on behalf of the USEPA(1991)	• Identified VOCs, PAHs, PCBs, and metals at concentrations greater than background in surface and subsurface soil samples at identified locations	• Investigation limited to nine surface samples and two subsurface samples • No groundwater analysis • Sample locations are approximate
Focus Site Inspection (FSI) including Site inspection, review of available information	PRC Environmental Management, Inc. (PRC), Focused Site Inspection Prioritization Site Evaluation Report for South Dayton Dump (1995)	• Evaluated potential threat to human health and the environment based on available information • Recommended the installation and sampling of groundwater monitoring wells and collection and analysis of surface water samples	• No sampling was conducted during the 1995 FSIP
Collected soil and groundwater samples for lithologic description, field screening and VOC analysis. Installed three permanent groundwater monitoring wells.	PSARA Technologies, Inc. (PSARA), Installation of Groundwater Monitoring Wells at the South Dayton Dump, Moraine Ohio. Prepared on behalf of Ohio EPA (1996)	• Identified the presence of methane in the sample headspace at five boring locations • Identified VOCs in concentrations that were less than MCLs but greater than Tapwater criteria.	• Investigation limited to seven locations • Three permanent monitoring wells were installed without previous VAS investigations to determine the depth of greatest groundwater contamination • No stratigraphic log is available for DP&L monitoring well (MW-104), which was sampled as a background location

DATA TYPE	*SOURCE*	*DATA USES RELATIVE TO THE CURRENT PROJECT*	*FACTORS AFFECTING THE RELIABILITY OF DATA AND LIMITATIONS ON DATA USE*
Collection and analysis of soil, sediment and groundwater samples.	Ohio EPA, Site Team Evaluation Prioritization Report, South Dayton Dump and Landfill (1996)	• Ohio EPA concluded human health soil, groundwater, sediments and surface water and air exposure pathways were all potentially complete • Report noted that the presence of PAHs in some of the samples could be attributed to the Valley Asphalt plant	• Evaluation of site-related contaminants is somewhat qualitative, as no statistical evaluation of background soil and water quality was completed • Unknown sample collection methods
Site Investigations including groundwater monitoring well installations, groundwater sampling and analysis and water level measurements	Payne Firm, Inc. (PFI), Groundwater monitoring well installations, groundwater sampling, analyses, and water level measurements (1998-2005)	• Collected ten rounds of groundwater samples and analyzed for VOCs, metals and Natural Attenuation Indicators • Confirmed the presence of chlorinated solvents and inorganic chemicals at the Site • Concluded that natural degradation of VOCs was occurring at the Site • Collected and analyzed surface water and sediment samples at the Quarry Pond in 1999 and 2000 for VOCs and TOC. • None of the surface water or sediment samples contained detectable concentrations of VOCs	• PFI noted that seasonal fluctuations in water table depth often result in variations in groundwater flow direction(s) and hence may affect groundwater quality at a given monitoring well location at a particular time, which was not supported by repeated sample events scheduled to coincide with variations in flow direction. • Target Compound List (TCL) analysis was not completed on the groundwater samples
Composite waste sample analysis on soil and drummed wastes	TCA Environmental (TCA), Environmental Remediation Report at Valley Asphalt. Prepared for Valley Asphalt (2000)	• Five drums containing a solid material were removed. The material removed was classified as a "characteristic hazardous waste", pursuant to RCRA and 40 CFR Part 261 Subpart C, (lead and cadmium) with PCBs, and disposed of at the Clean Harbors facility in Cincinnati, Ohio. • One drinking water well and one production well were located in the vicinity of the excavated area	• The TCA report does not describe the condition of the excavation prior to backfilling. However, CRA spoke with Dale Farmer, Ohio EPA's On-Scene Coordinator (OSC) on December 15, 2006 who advised that the drums encountered had been crushed prior to excavation, and that there was a corner of a drum and other debris visible in the side wall of the excavation.

QAPP Worksheet #14 - Summary of Project Tasks

SAMPLING AND ANALYSIS TASKS	
Sampling Tasks:	See Data Quality Objectives Tables, Worksheet #11-1 to Worksheet #11-6.
Analysis Tasks:	• Water (Groundwater, Surface water): VOC 5030B/8260B, SVOC 3520/8270, Pesticides 3520C/8081A, PCB 3520C/8082, Metals 3051/6010B/200.7, Mercury 7470A, Herbicides 8151A, Cyanide 9012A/335.2, 335.4, Alkalinity 310.1/2320B, Anions 300/9056A, Sulfide 9030B/9034, pH 150.1/4500H+B/9040, • Soil and Fill (Surfical, Sub-surface, Sediment): VOC 5035/8260C, SVOC 3540/8270, Pesticides 3540C/8081A, PCBs 3540C/8082, Metals 3050/6010, Mercury 7471, Herbicides 8151A, Cyanide 9012A, Sulfide 9030B/9034, pH 9045 • Vapor intrusion Activities (Soil Gas. Sub-slab, Indoor air, Outdoor air, Crawl space, if necessary): TCL VOCs/TO15_OH • Waste characterization (Soil cutting, Purged Groundwater): VOC 5030B/8260B, SVOC 3520/8270, Pesticides 3520C/8081A, PCB 3520C/8082, Metals 3051/6010B/200.7, Mercury 7470A, Herbicides 8151A, Cyanide 9012A/335.2, 335.4, Sulfide 9030B/9034, pH 150.1/4500H+B/9040, Flashpoint 1010
Quality Control Tasks:	The samples will be collected and processed, and the laboratory waste will be disposed of as described in the laboratory SOPs (*Laboratory Sample Analysis SOPs-Appendix D, Laboratory Sample Preparation SOPs-Appendix E, and Laboratory Support SOPs-Appendix F).* QA samples will be collected as described in Worksheet #20. All the following QC checks will be performed as applicable to the specific method: tuning, initial calibration, continuing calibration checks, laboratory control samples (LCS), surrogates, method blanks, instrument blanks, and all other applicable QC as defined in the analytical methods.
Secondary Tasks:	See Worksheets #13

DATA MANAGEMENT, DOCUMENTATION, RECORDS, AND AUDIT TASKS	
Data Management Tasks:	**Field data reduction –** Raw data from field measurements and sample collection activities will be recorded as specified in the Field Sampling Plan (CRA, 2013). Only direct-reading instrumentation will be employed in the field. The CRA Project Manager or designee will proofread all forms and notebooks to determine if transcriptionerrors have been made by the field crew. **Laboratory data reduction –** TestAmerica-North Canton (TA-NC) and TestAmerica-Knoxville (TA-KX) will perform in-house analytical data reduction under the direction of the laboratory QA/QC Managers. The laboratory QA/QC Managers will be responsible for assessing data quality and advising of any data that were rated "preliminary" or "unacceptable" or of other notations that would caution the data user of possible unreliability. Data reduction, by the laboratory, will be conducted as follows: • The analysts who produced the laboratory data will first conduct a systematic review (Level 1 Review). • An experienced peer, supervisor, or designee will examine the data (the Level 2 Review) to ensure that the Level 1 review has been completed correctly and thoroughly. Following the Level 2 review, the data will be turned over to the Laboratory Project Manager for a third-level review. • The Project Manager will review the data for completeness and attainment of quality control criteria as outlined in the USEPA methods and for overall reasonableness. • The Project Manager will verify the accuracy and completeness of the final reports. • The Laboratory QA/QC Manager and the supervisor of the pertinent analytical section, in conjunction with the CRA QA Officer, will decide whether any sample reanalysis is required. Data reduction procedures are included in the USEPA-approved methods and associated laboratory SOPs **Field data reporting –** Field data reporting will consist of field logs and calibration and measurement records documenting site activities as described in the Field Sampling Plan (CRA, 2013) and on the sample Chain-of-Custody (COC) Records.

DATA MANAGEMENT, DOCUMENTATION, RECORDS, AND AUDIT TASKS	
Data Management Tasks: ***(continued)***	**Laboratory data reporting –** The analytical laboratories will prepare and retain full analytical and QC documentation. Such retained documentation need not be on hard (paper) copy, but may be in other storage media (*e.g.*, computer diskette or magnetic tape). As needed, TA-NC, TA-KX, or affiliate laboratory will supply a hard copy of the retained information. TA-NC, TA-KX, or affiliate laboratory will provide the following information in each analytical data package submitted: • Dated cover sheets, signed by the TA-NC, TA-KX or affiliate laboratory Project Manager, listing a laboratory batch number; the analyses performed; the number of samples and respective matrices; the project name and number; narrative comments describing deviations from intended analytical strategy, and any problems encountered in analysis; a discussion of any laboratory quality control checks that failed to meet project criteria; and the signature of the laboratory QA/QC Manager. • Tabulated results of inorganic and organic compounds identified and quantified, including sample preparation and analysis dates, and cross-references of laboratory and field sample identification numbers. • Analytical results for QC sample spikes and sample duplicates; initial and continuing calibration verifications of standards and blanks; standard procedural blanks; laboratory control samples; and the data produced by ICP interference check samples, as appropriate for the specified analyses. • Tabulation of Method Detection Limits, as appropriate. • Raw data system printouts (or legible photocopies) identifying the date of analyses, the mass spectra tuning data, the name of the analyst, the parameters determined, the initial and continuing calibration, the calibration verification summary, the method blanks, the sample and any dilutions, sample duplicates and spikes, chromatograms, GC/MS spectra, computer printouts, internal standard area and RT summary, cleanup information, control samples, ICP outputs, and inter-element correction data.

DATA MANAGEMENT, DOCUMENTATION, RECORDS, AND AUDIT TASKS	
Documentation and records:	A report will be prepared containing a QA/QC section summarizing the quality of the data. The QA report prepared by CRA will address the assessment of data precision, accuracy, completeness, and comparability; the results of performance audits, if any; the results of system audits; any reported non-conformances; any significant QA/QC problems and recommended solutions; the results of corrective actions since the last report; and approved revisions for the intended purposes based on an evaluation of compliance with control limits, the results of audits, and compliance with the procedures specified in the QAPP and the FSP. The report will indicate whether the QA objectives were met and whether the data can be used. Appropriate records will be maintained to provide adequate documentation of the entire data generation process, including field sampling and laboratory analysis: **Field documentation –** Field personnel will develop and retain comprehensive records of field activities, including field sampling, field analysis, and sample COC Record, to allow a reconstruction of field events and sample handing during data review and interpretation. **Laboratory project files –** TA-NC, TA-KX or affiliate laboratory will maintain a file for pertinent project information, including COC Records; other custody documents (air bills, etc.); work orders; Sample Receipt Acknowledgment Forms, if any; instrument detection limit and control limit tabulations; all raw analytical data on bench sheets; laboratory data; and project communication records. Such retained documentation need not be on hard (paper) copy, but may be in other storage media (*e.g.*, computer diskette or magnetic tape). As needed, TA-NC, TA-KX or subcontractor laboratory will supply a hard copy of the retained information. **Laboratory notebooks –** Logbooks, bench sheets, instrument notebooks, and instrument printouts will be retained as part of the permanent laboratory record, including the associated quality controls. Each page in the laboratory logbooks and bench sheets will be signed and dated by the analyst, and errors will be crossed out in indelible ink. System printouts of raw inorganic and organic data will include dates of analyses; analyst's name; parameters determined; calibration curve; calibration verifications; method blanks; sample number and dilutions performed; and sample duplicates, spikes, and control samples. Internal laboratory QC sample results will be indicated on the analytical bench sheets and will include sample spikes, sample duplicates, initial and continuous calibration verification of standards and blanks, standard procedural blanks, laboratory control samples, ICP serial dilutions, and ICP interference check samples.

DATA MANAGEMENT, DOCUMENTATION, RECORDS, AND AUDIT TASKS	
Documentation and records: *(continued)*	**Computer and hard copy storage –** All electronic files and deliverables will be retained by the laboratory for no less than 5 years. TestAmerica Laboratories or its designated representatives will retain copies of the analytical data reports according to the requirements of the laboratory QA Manual. All field records will be kept in the central project file at the CRA offices at Niagara Falls, Waterloo, or Cincinnati, and in electronic files; and records will be included in project reports, as appropriate or upon request by the USEPA RPM. Records will be reviewed by the CRA Project Manager for consistency with the planned activities, and any concerns will be discussed with the Field QA Officer. Field performance and field system audits will also be performed, as discussed below and in Worksheets #31 and 32. **Laboratory data reporting –** Analytical data for this project will be reported in both an electronic data deliverable (EDD) and an analytical data package. The EDD will be generated by TA-NC, TA-KX or affiliate laboratory and will be used by CRA to facilitate loading the analytical data into the project database. The Laboratory QA/QC Manager will perform a final review of the report summaries and case narratives to determine if the report meets project requirements. The task of reporting laboratory data to the USEPA will begin after the data review activity has been concluded. The validated analytical data will be provided to the USEPA in accordance with the project schedule (Worksheet #16). In addition to the COC Record, TA-NC , TA-KX or affiliate laboratory: • **Case narrative –** Date of issuance; laboratory analysis performed; any deviations from required analytical methods; laboratory sample lot numbers; numbers of samples and respective matrices; QC procedures used and references to the acceptance criteria; laboratory report table of contents; project name and number; condition of samples upon receipt; dates of extraction, preparation, and analysis; discussion of whether or not sample holding times were met; discussion of technical problems or other observations that may have created analytical difficulties; discussion of any laboratory QC checks that failed to meet project criteria; signature of the laboratory Project Manager, and copies of the COC Records • **Chemistry data package –** Run log, summary page indicating dates of analyses for samples and laboratory QC checks, cross-referencing of laboratory sample to project sample identification numbers**,** adequately described data qualifiers, sample preparation and analysis methods, sample results, matrix spike and matrix spike duplicate (MS/MSD) recoveries, laboratory control sample recoveries, method blank results, and surrogate recoveries

DATA MANAGEMENT, DOCUMENTATION, RECORDS, AND AUDIT TASKS	

Documentation and records: ***(continued)***	Groundwater data will be reported in micrograms per liter (µg/L). Results between the laboratory Method Detection Limit (MDL) and the Quantitation Limit (QL) will be reported. Data retained in the project database may be converted to units other than those reported by the laboratories. Sample results will not be corrected for contamination found in laboratory blanks. However, sample results may be qualified as not detected based on laboratory, field, and/or trip blank contamination.
	
	TA-NC, TA-KX or affiliate laboratory will provide the following information in each analytical data package submitted:
	
	• Dated cover sheets, signed by the TA-NC, TA-KX or affiliate laboratory Project Manager, listing a laboratory batch number; the analyses performed; the number of samples and the respective matrices; the project name and number; narrative comments describing deviations from intended analytical strategy, and problems encountered in analysis; a discussion of any laboratory quality control checks that failed to meet project criteria; and the signature of the laboratory QA Manager
	• Tabulated results of inorganic and organic compounds identified and quantified, including sample preparation and analysis dates, and cross-references of laboratory and field sample identification numbers
	  • Analytical results for QC sample spikes, sample duplicates, and blanks; initial and continuing calibration verifications of standards and blanks; standard procedural blanks; laboratory control samples; and the data produced by inductively coupled plasma (ICP) interference check samples, as appropriate for the specified analyses
	  • Tabulation of Method Detection Limits, as appropriate
	  • Raw data system printouts (or legible photocopies) identifying dates of analyses, mass spectra tuning data, name of analyst, parameters determined, initial and continuing calibration, calibration verification summary, method blanks, sample and any dilutions, sample duplicates and spikes, chromatograms, gas chromatograph/mass spectrophotometer (GC/MS) spectra, computer printouts, internal standard area and retention time (RT) summary, cleanup information, control sample results, ICP outputs, and inter-element correction data

DATA MANAGEMENT, DOCUMENTATION, RECORDS, AND AUDIT TASKS	
Assessment/Audit Tasks	Performance and system audits will be completed in the field and laboratory, as described below and in Worksheets #31 and 32. **Field audits –** The Project Manager will monitor day-to-day field performance through daily communications with the on-site field staff. In addition, field performance audits and field system audits will be performed, as follows: • **Field performance audits –** Field performance audits will be conducted in order to confirm that the activities are being performed according to the established plans. The field performance audit(s) will be performed by the Senior Consultant QA Manager (or his/her designee), at a frequency that is appropriate for the field activities being performed. The audit(s) will include a discussion of the project progress with the Project Manager and/or the review of field reports, as appropriate. The Senior Consultant QA Manager will record and document any observations made during field system audits, and will discuss the audit and any recommended changes/deviations to the field procedures with the Project Manager. • **Field system audits –** Field system audits will be performed by the CRA QA Officer, including a review of rinse and trip blank data to identify potential deficiencies in field sampling and decontamination procedures, and a comparison of the scheduled QA/QC activities described in this QAPP with the QA/QC procedures being performed on the project. Field system audits will be performed at a frequency appropriate for the field activities. The CRA QA Officer will record and document any observations made during field system audits, and will discuss the audit and any recommended changes/deviations to the field procedures with the Project Manager. **Laboratory audits –** Laboratory audits will be performed, as follows: • **Internal audits –** The Laboratory QA/QC Manager (or his/her designee) will conduct internal laboratory audits periodically. This will include an overall evaluation of the performance of laboratory staff and a comparison of laboratory procedures with the laboratory QA Manual and SOPs. Results of the audits will be summarized and distributed to appropriate laboratory staff. • **External audits –** The CRA QA Officer will review the laboratory QA Manual and applicable SOPs, and will discuss laboratory procedures with the Laboratory QA/QC Manager prior to the start of project sampling. The CRA QA Officer will record and document any observations made during the review. In addition, as a participant in state and federal certification programs, the laboratory is audited by representatives of the regulatory agency issuing certification. Audits include a review of sample handling and tracking documentation, analytical methodologies, analytical supportive documentation, and final reports. The audit findings are documented and submitted to the laboratory for corrective action, if necessary.

DATA MANAGEMENT, DOCUMENTATION, RECORDS, AND AUDIT TASKS	
Assessment/Audit Tasks ***(continued)***	**Corrective action –** Corrective actions are required when field or analytical data are not within the objectives specified in this QAPP, as follows: • **Field measurement corrective action –** Corrective action in the field may be necessary when the sample network is changed (*i.e.* more/fewer samples, sampling locations other than those specified in the FSP, etc.), or when sampling procedures and/or field analytical procedures require modification in response to unexpected conditions. Technical staff and project personnel will be responsible for reporting all suspected technical or QA non-conformances or deficiencies of any activity or issued document by reporting the situation to the CRA Project Manager or designee. The CRA Project Manager will assess the suspected problems in consultation with the CRA QA Officer or designee, and will assist in making a decision based on the potential for the situation to impact the data quality. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, the CRA Field QA Officer will initiate a nonconformance report. If appropriate, the CRA Field QA Officer will ensure that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed. • **Laboratory corrective action –** Corrective actions are required whenever an out-of-control event or potential out-of control event is noted. Corrective actions may be necessary if any of the following occur: - QC data are outside the warning or acceptable windows for precision and accuracy - Blanks contain target analytes above acceptable levels. - Undesirable trends are detected in spike recoveries or the RPD between duplicates - There are unusual changes in detection limits - Deficiencies are detected by the Laboratory Manager during internal or external audits or from the results of performance evaluation samples - Inquiries concerning data quality are received. Corrective actions should be timely, and they should determine the root cause and evaluate any propagation of the error or problem. The investigative action taken is somewhat dependent on the analysis and the event. Corrective action in the laboratory may occur prior to, during, or after the initial analysis. Corrective action is under the supervision of the Laboratory Project Manager and Laboratory QA Officer. Following a consultation with laboratory scientists, technicians, and team leaders, it may be necessary for the Laboratory Manager to approve the implementation of the corrective action. Some conditions during or after analysis may automatically trigger corrective action or optional procedures. These conditions may include dilution of samples, additional sample extract cleanup, and automatic reinjection/reanalysis when certain quality control criteria are not met. TA-NC, TA-KX or affiliate laboratory corrective action procedures are documented in Laboratory SOPs specifying corrective action to be taken when an analytical error is discovered or the analytical system is found to be out of control.

DATA MANAGEMENT, DOCUMENTATION, RECORDS, AND AUDIT TASKS	
Assessment/Audit Tasks ***(continued)***	Depending on the problem, the corrective action employed may be formal or informal. On-the-spot actions are used to correct minor problems, such as recalibration, retuning, or a minor repair (*e.g.*, replacement of a minor part) of a malfunctioning instrument or the correction of poor analytical technique being used. Corrective action procedures may be handled at the bench level by the analyst, who reviews the preparation or extraction procedure that was used for possible errors, and checks the instrument calibration, spike, and calibration mixes, and the instrument sensitivity. These occurrences are documented in the appropriate injection, run, or analysis logbooks. Similarly, routine instrument maintenance, malfunctions, and power failures are also documented in the appropriate instrument maintenance logbooks. If the problem persists or cannot be identified, the matter may be referred to the laboratory team leader, and/or QA/QC Manager for further investigation. Occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated will be properly documented. The corrective action procedure will be discussed with the Laboratory Project Manager, and full documentation of the corrective action procedure, whether resolved or not, will be placed in the laboratory project file. Corrective actions specific to analytical methods are discussed in the operational-specific SOPs. The USEPA RPM or the CRA QA Officer may request corrective action for any nonconformance identified by audits or data validation. **Corrective action during data validation and data assessment –** The need for corrective actions may be identified during data validation or data assessment. Potential types of corrective action may include re-sampling by the field team or reinjection/reanalysis of samples by the laboratory. Data validation corrective actions may include notification of the laboratory of incomplete or erroneous reports and a request for issuance of corrected versions. When the CRA QA Officer identifies a corrective action situation, the CRA Project Manager will approve the implementation of corrective action, including possible re-sampling. The CRA QA Officer will notify the laboratory of incomplete or erroneous reports and will request the issuance of corrected versions. All corrective actions will be documented. Final summary data tables will not be issued until all data have been validated and all corrections have been made. Corrective action may include the following: • Reanalysis of samples, if holding time requirements permit • Re-sampling and analysis • Evaluation and amendment of sampling procedures • Evaluation and amendment of analytical procedures
Data review tasks:	See Worksheets #36 and 37.

TABLE 15.1

SOIL SCREENING LEVELS
QUALITY ASSURANCE PROJECT PLAN
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

	Project Action Limits		Project Action Limits		Quantitation Limit	Method Detection Limit
	USEPA Regional Screening Levels (RSLs) [1]		*Ecological Screening Levels* [2]	*Ecological Screening Values* [3], [4]		
	Residential Soil	*Industrial Soil*				
	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
Parameter						
Volatile Organic Compounds						
Chlorobenzene	28,000	130,000	13,100	50	5	0.33
Ethylbenzene	5,800	25,000	5,160	50	5	0.26
Tetrachloroethene	8,100	39,000	9,920	10	5	0.52
Vinyl chloride	59	1,700	646	10	5	0.39
Semi-Volatile Organic Compounds						
Benzo(a)anthracene	150	2,900	5,210	-	6.67	0.63
Benzo(a)pyrene	15	290	1,520	100	6.67	0.64
Benzo(b)fluoranthene	150	2,900	59,800	-	6.67	0.59
Benzo(k)fluoranthene	1,500	29,000	148,000	-	6.67	0.68
Dibenz(a,h)anthracene	15	290	18,400	-	6.67	0.66
Indeno(1,2,3-cd)pyrene	150	2,900	109,000	-	6.67	0.35
Naphthalene	3,800	17,000	99.4	100	6.67	0.82
Metals						
Antimony	3,100	47,000	142	3,500	1,000	390
Arsenic	670	3,000	5,700	10,000	1,000	300
Cobalt	2,300	35,000	140	20,000	5,000	160
Copper	310,000	4,700,000	5,400	40,000	2,500	740
Iron	5,500,000	82,000,000	-	200,000	10,000	4900
Lead	400,000	800,000	53.7	50,000	300	190
Manganese	180,000	2,600,000	-	100,000	1,500	74
PCBs						
Aroclor-1016 (PCB-1016)	400	5,200	-	-	33	21
Aroclor-1221 (PCB-1221)	150	660	-	-	33	16
Aroclor-1232 (PCB-1232)	150	660	-	-	33	14
Aroclor-1242 (PCB-1242)	240	1,000	-	-	33	13
Aroclor-1248 (PCB-1248)	240	1,000	-	-	33	17
Aroclor-1254 (PCB-1254)	110	1,000	-	-	33	17
Aroclor-1260 (PCB-1260)	240	1,000	-	-	33	17
Total PCBs	-	-	0.332	20	-	-
Pesticides						
Dieldrin	33	140	2.38	0.5	1.7	0.47

Notes:

- - Not applicable.

[1] - United States Environmental Protection Agency Regional Screening Levels (RSL), May 2014, based on Target Cancer Risk of 1E-06, and Total Hazard Quotient of 0.1.

[2] - United States Environmental Protection Agency RCRA Ecological Screening Levels, August 22, 2003

[3] - G.P. Friday, 1998. Ecological Screening Values for Surface Water, Sediment, and Soil. Wesinghouse Savannah River Company. Report WSRC-TR-98-00110

[4] - USEPA. 2001. Supplemental Guidance to RAGS: Region 4 Bulletins, Ecological Risk Assessment. Originally published November 1995. Website version last updated November 30, 2001: http://www.epa.gov/region4/superfund/programs/riskassess/ecolbul.html

ID	Task Name	Duration	Start	Finish	Predecessors
1	**SHALLOW GROUNDWATER INVESTIGATION AND DELIVERABLES [1]**	**507 days**	**Mon 2/4/13**	**Tue 1/13/15**	
2	**Phase 1A - Shallow Groundwater Data Gap Investigation [1]**	**345 days**	**Mon 2/4/13**	**Fri 5/30/14**	
3	Submit Draft Work Plan	41 days	Mon 2/4/13	Mon 4/1/13	
4	USEPA Review and Comment	21 days	Tue 4/2/13	Tue 4/30/13	3
5	Submit Final Work Plan and USEPA approval	1 day	Fri 5/10/13	Fri 5/10/13	4
6	Field Work: Data Gap Area Investigation	23 days	Mon 6/10/13	Wed 7/10/13	5FS+10 days
7	Summarize results; Propose Locations for Phases 1B and 2A investigations with rationale	31 days	Fri 8/9/13	Fri 9/20/13	6FS+10 days
8	In-person Meeting regarding next steps for developing workplans and data collection	1 day	Tue 7/23/13	Tue 7/23/13	
9	Conference call to discuss results and proposed locations	1 day	Thu 9/5/13	Thu 9/5/13	
10	USEPA Review and Comment	10 days	Thu 10/17/13	Wed 10/30/13	7
11	Submit Revised Phase 1A Summary and Proposal for Phase 1B and Phase 2A	57 days	Thu 10/31/13	Fri 1/17/14	10
12	USEPA Review and Comment, and Approval of Proposed Locations	65 days	Mon 3/3/14	Fri 5/30/14	11
13	**OU1 RI/FS Report**	**121 days**	**Mon 7/7/14**	**Mon 12/22/14**	
14	Submit Draft Report	60 days	Mon 7/7/14	Fri 9/26/14	
15	USEPA Review and Comment	22 days	Mon 9/29/14	Tue 10/28/14	14
16	Conference call to discuss USEPA comments on OU1 RI/FS Draft Report	1 day	Wed 10/29/14	Wed 10/29/14	15
17	Submit Final RI/FS Report	22 days	Wed 10/29/14	Thu 11/27/14	15
18	USEPA Approval	17 days	Fri 11/28/14	Mon 12/22/14	17
19					
20					
21	**OU2 RI/FS Work Plan**	**187 days**	**Thu 9/12/13**	**Fri 5/30/14**	
22	Submit Draft Report to USEPA	1 day	Thu 9/12/13	Thu 9/12/13	
23	USEPA Review and Comment	51 days	Fri 9/13/13	Fri 11/22/13	22
24	Conference call to discuss USEPA comments on OU2 RI/FS Work Plan	1 day	Thu 12/12/13	Thu 12/12/13	23
25	Submit Revised Work Plan	50 days	Mon 11/25/13	Fri 1/31/14	23
26	USEPA Review and Comment	48 days	Mon 2/3/14	Wed 4/9/14	25
27	Submit Revised Work Plan	15 days	Thu 4/10/14	Wed 4/30/14	26
28	USEPA Review and Comment and Approval	20 days	Thu 5/1/14	Wed 5/28/14	27
29	Submit updated Quality Assurance Project Plan (QAPP)	1 day	Fri 5/30/14	Fri 5/30/14	27FS+21 days
30					
31	**OU2 REMEDIAL INVESTIGATIONS AND DELIVERABLES**	**285 days**	**Mon 6/23/14**	**Fri 7/24/15**	
32	**OU2 Soil Investigation**	**115 days**	**Mon 6/23/14**	**Fri 11/28/14**	
33	Field Work: OU2 Phase 1A & 1B Soil Investigation	19 days	Mon 6/23/14	Thu 7/17/14	28FS+13 days
34	Summarize results	20 days	Fri 8/1/14	Thu 8/28/14	33FS+10 days
35	Conference call to discuss results and proposed additional locations	1 day	Fri 8/15/14	Fri 8/15/14	34SS+10 days
36	Submit summary of OU2 Soil Phases 1A & 1B results, and proposal for OU2 Soil Phase 2 investigative locations (if necessary)	1 day	Fri 8/29/14	Fri 8/29/14	34,35
37	USEPA Review and Comment	21 days	Mon 9/1/14	Mon 9/29/14	36
38	Submit Revised OU2 Soil Investigation Summary, and Phase 2 Proposal (if necessary)	15 days	Tue 9/30/14	Mon 10/20/14	37
39	USEPA Review and Approval	10 days	Tue 10/21/14	Mon 11/3/14	38
40	Field Work: OU2 Phase 2 Soil Investigation	10 days	Mon 11/17/14	Fri 11/28/14	39FS+9 days
41	**OU2 SURFACE WATER AND SEDIMENT INVESTIGATIONS**	**265 days**	**Mon 7/21/14**	**Fri 7/24/15**	
42	Field Work: OU2 Surface Water and Sediment Investigation (1st sampling event)	10 days	Mon 7/21/14	Fri 8/1/14	28FS+37 days
43	Field Work: OU2 Surface Water Investigation (2nd sampling event)	10 days	Mon 4/13/15	Fri 4/24/15	42FS+180 days
44	Summarize results	20 days	Mon 5/11/15	Fri 6/5/15	43FS+10 days
45	Submit Draft OU2 Surface Water and Sediment Investigation Summary Letter Report and propose additional investigation locations (if necessary)	1 day	Fri 6/5/15	Fri 6/5/15	44FF
46	USEPA Review and Comment	20 days	Mon 6/8/15	Fri 7/3/15	45
47	Submit Final OU2 Surface Water and Sediment Investigation Summary Letter Report	15 days	Mon 7/6/15	Fri 7/24/15	46
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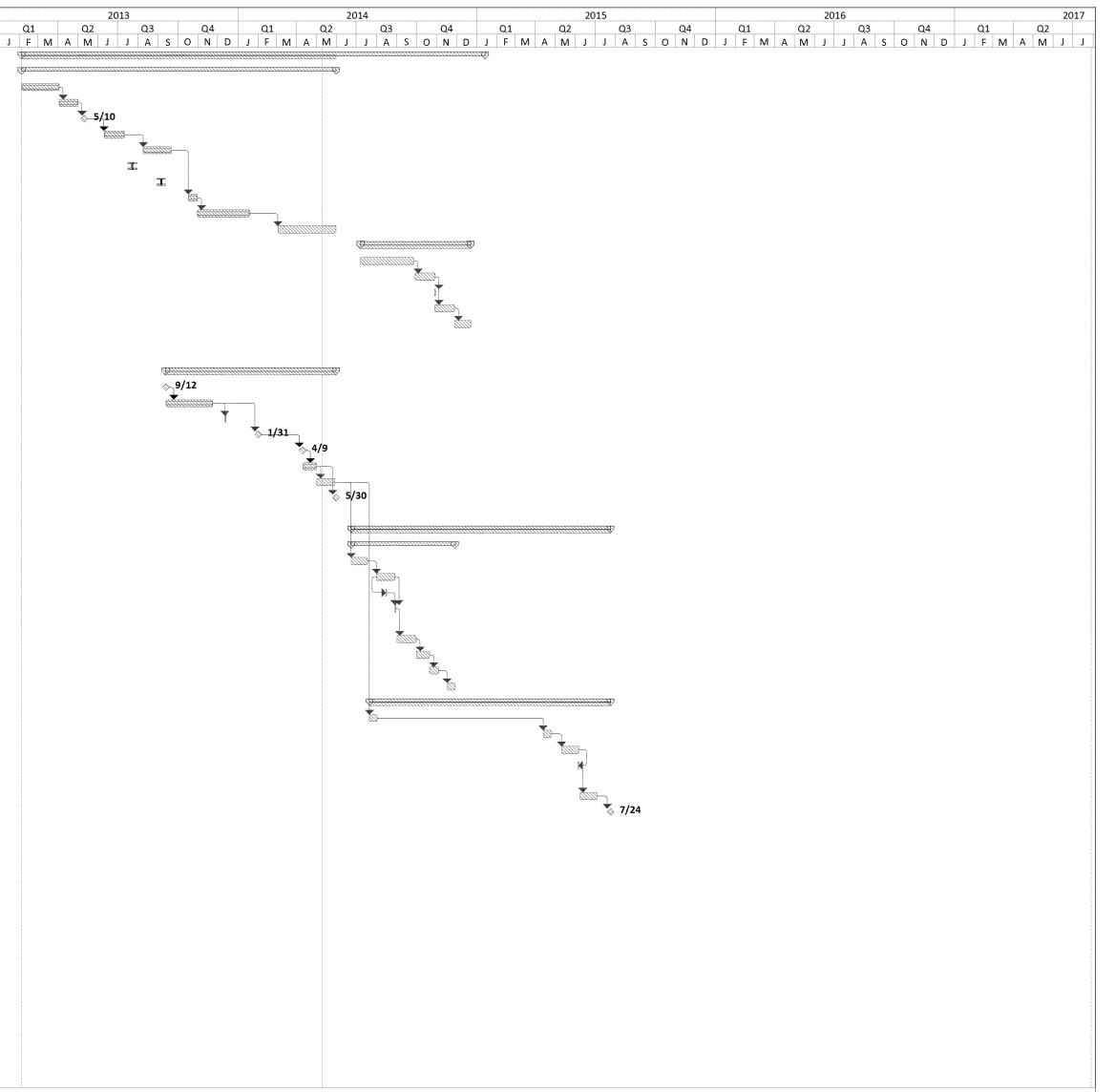

Project: USEPA_OU2 RI-FS draftSc
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Task | Summary | External Milestone | Inactive Summary | Manual Summary Rollup | Finish-only
Split | Project Summary | Inactive Task | Manual Task | Manual Summary | Deadline
Milestone | External Tasks | Inactive Milestone | Duration-only | Start-only | Progress

[1] - As required by the Dispute Resolution, dated December 15, 2010
[2] - Corresponds to Phase 2A or Phase 2B, detailed in the Final Work Plan for Operable Unit One (OU1) Groundwater and Data Gap Investigation - Phase 1A (Work Plan), dated May 10, 2013

ID	Task Name	Duration	Start	Finish	Predecessors
65					
66	**USEPA issuance of a new ASAOC_1**	**0 days**	**Mon 9/1/14**	**Mon 9/1/14**	
1	USEPA issuance of a new ASAOC	0 days	Mon 9/1/14	Mon 9/1/14	
67	**PRELIMINARY OU2 INVESTIGATIONS AND DELIVERABLES [2]**	**270 days**	**Tue 9/16/14**	**Mon 9/28/15**	**66**
68	**Phase 2A - OU2 VAS GW Investigation [2]**	**70 days**	**Tue 9/16/14**	**Mon 12/22/14**	
69	Field Work: Advance VAS Boreholes	25 days	Tue 9/16/14	Mon 10/20/14	66FS+10 days
70	Summarize results	25 days	Tue 11/4/14	Mon 12/8/14	69FS+10 days
71	Conference call to discuss results and proposed additional locations	1 day	Tue 11/18/14	Tue 11/18/14	70SS+10 days
72	Submit proposal for Phase 2B and further VAS investigation locations (if required)	1 day	Mon 12/8/14	Mon 12/8/14	70FF,71
73	USEPA Review, Comment and Approval of Proposed Locations	10 days	Tue 12/9/14	Mon 12/22/14	72
74	**Phase 1/2B - Permanent Well installation [2]**	**150 days**	**Tue 3/3/15**	**Mon 9/28/15**	
75	Field Work: Installation of Permanent Wells	15 days	Tue 3/3/15	Mon 3/23/15	73FS+50 days
76	Field Work: Development and sampling newly installed permanent wells (1st sampling event)	10 days	Tue 3/17/15	Mon 3/30/15	75SS+10 days
77	Summarize results of 1st GW sampling event	15 days	Tue 4/14/15	Mon 5/4/15	76FS+10 days
78	Field Work: Sample newly installed monitoring wells (2nd sampling event)	10 days	Tue 7/21/15	Mon 8/3/15	76FS+80 days
79	Summarize results of 2nd GW sampling event	20 days	Tue 8/18/15	Mon 9/14/15	78FS+10 days
80	Conference call to discuss results and proposed locations	1 day	Tue 9/1/15	Tue 9/1/15	79SS+10 days
81	Submit proposal for further investigative locations (if required)	1 day	Mon 9/14/15	Mon 9/14/15	79FF
82	USEPA Review and Comment and Approval of Proposed Locations (if required)	10 days	Tue 9/15/15	Mon 9/28/15	81
83	**OU2 REMEDIAL INVESTIGATIONS AND DELIVERABLES**	**713 days**	**Wed 11/5/14**	**Fri 7/28/17**	
84	**OU2 GROUNDWATER INVESTIGATION**	**281 days**	**Tue 6/2/15**	**Tue 6/28/16**	
85	Submit OU2 GW Letter Work Plan (based on results of OU2 Soil Phases 1A & 1B Investigations)	1 day	Tue 6/2/15	Tue 6/2/15	77FS+20 days
86	USEPA Review and Comment	20 days	Wed 6/3/15	Tue 6/30/15	85
87	Conference call to discuss USEPA Comments on Draft OU2 GW Letter Work Plan	1 day	Mon 7/6/15	Mon 7/6/15	86FS+3 days
88	Submit revised OU2 GW Letter Work Plan	15 days	Wed 7/1/15	Tue 7/21/15	86
89	USEPA Review and Approval of Final OU2 GW Letter Work Plan	10 days	Wed 7/22/15	Tue 8/4/15	88
90	Field Work: OU2 GW Investigation	15 days	Wed 8/19/15	Tue 9/8/15	89FS+10 days
91	Field Work: OU2 GW monitoring well sampling (1st sampling event)	10 days	Wed 9/16/15	Tue 9/29/15	90FS+5 days
92	Field Work: OU2 GW monitoring well sampling (2nd sampling event)	10 days	Wed 3/16/16	Tue 3/29/16	91FS+120 days
93	Summarize results	20 days	Wed 4/13/16	Tue 5/10/16	90FS+10 days,9
94	Submit OU2 GW Investigation Summary Letter Report and propose additional investigation locations (if necessary)	1 day	Wed 5/11/16	Wed 5/11/16	93
95	USEPA Review and Comment	20 days	Thu 5/12/16	Wed 6/8/16	94
96	Submit Final OU2 GW Investigation Summary Letter Report	14 days	Thu 6/9/16	Tue 6/28/16	95
97	**OU2 SOIL GAS INVESTIGATIONS (IF NECESSARY)**	**154 days**	**Mon 11/16/15**	**Thu 6/16/16**	
98	Submit OU2 Soil Gas Letter Work Plan (Based on results of OU2 GW and Soil Investigations)	1 day	Mon 11/16/15	Mon 11/16/15	91FS+33 days
99	USEPA Review and Comment	20 days	Tue 11/17/15	Mon 12/14/15	98
100	Conference Call to discuss USEPA Comments on Draft OU2 Soil Gas Letter Work Plan	1 day	Thu 12/17/15	Thu 12/17/15	99FS+2 days
101	Submit revised OU2 Soil Gas Letter Work Plan	15 days	Fri 12/18/15	Thu 1/7/16	100
102	USEPA Review and Approval of Final OU2 Soil Gas Letter Work Plan	20 days	Fri 1/8/16	Thu 2/4/16	101
103	Field Work: OU2 Soil Gas investigations	20 days	Fri 2/19/16	Thu 3/17/16	102FS+10 days
104	Summarize Results	20 days	Fri 4/1/16	Thu 4/28/16	103FS+10 days
105	Submit Draft OU2 Soil Gas Investigation Summary Letter Report and propose additional investigations (if necessary)	1 day	Thu 4/28/16	Thu 4/28/16	104FF
106	USEPA Review and Comment	20 days	Fri 4/29/16	Thu 5/26/16	105
107	Submit Final OU2 Soil Gas Investigation Summary Letter Report	15 days	Fri 5/27/16	Thu 6/16/16	106
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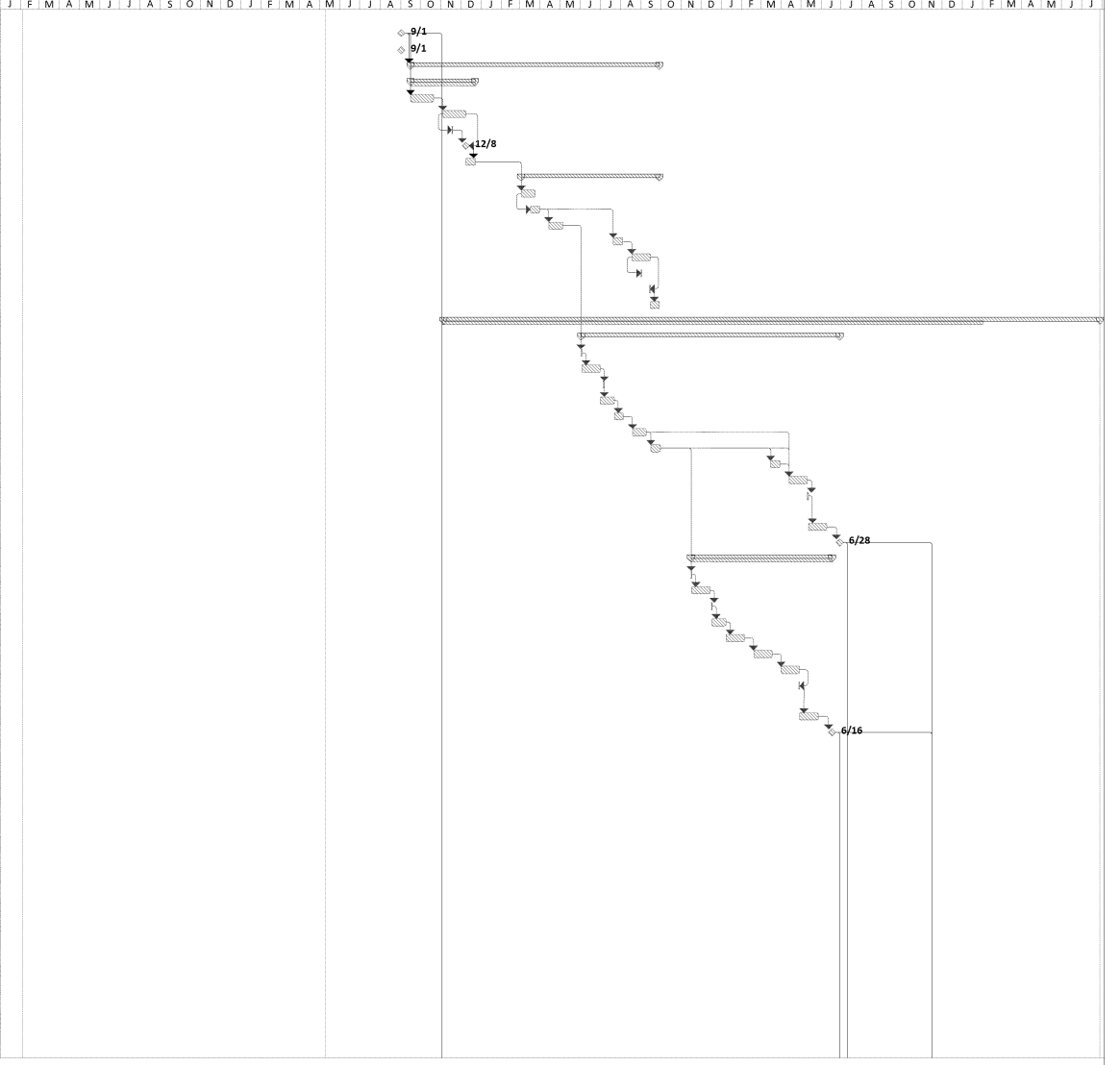

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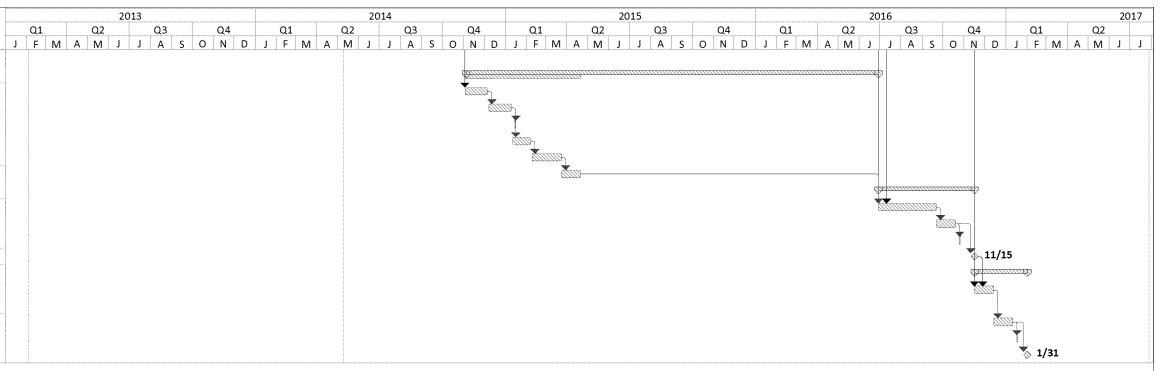
[2] - Corresponds to Phase 2A or Phase 2B, detailed in the Final Work Plan for Operable Unit One (OU1) Groundwater and Data Gap Investigation - Phase 1A (Work Plan), dated May 10, 2013

ID	Task Name	Duration	Start	Finish	Predecessors
128					
129	**RISK ASSESSMENT FRAMEWORK**	**430 days**	**Wed 11/5/14**	**Tue 6/28/16**	
130	Submit Risk Assessment Framework	25 days	Mon 11/3/14	Fri 12/5/14	66
131	USEPA Review and Comment	25 days	Mon 12/8/14	Fri 1/9/15	130
132	Conference Call to discuss USEPA Comments	1 day	Thu 1/15/15	Thu 1/15/15	131FS+3 days
133	Submit Revised Risk Assessment Framework	20 days	Mon 1/12/15	Fri 2/6/15	131
134	USEPA Review and Comment	31 days	Mon 2/9/15	Mon 3/23/15	133
135	Submit Final Risk Assessment Framework	20 days	Tue 3/24/15	Mon 4/20/15	134
136	**SCREENING LEVEL RISK ASSESSMENT**	**100 days**	**Wed 6/29/16**	**Tue 11/15/16**	
137	Submit Screening Level Risk Assessment Report	60 days	Wed 6/29/16	Tue 9/20/16	107,96,135
138	USEPA Review and Comment	20 days	Wed 9/21/16	Tue 10/18/16	137
139	Conference Call to discuss USEPA Comments	1 day	Tue 10/25/16	Tue 10/25/16	138FS+4 days
140	Submit Final Screening Level Risk Assessment Report	20 days	Wed 10/19/16	Tue 11/15/16	138
141	**OU2 RI/FS Summary Report**	**55 days**	**Wed 11/16/16**	**Tue 1/31/17**	
142	Submit OU2 RI/FS Summary Report (including RAO, data evaluation, screening of remedial technologies)	20 days	Wed 11/16/16	Tue 12/13/16	107,96,140
143	USEPA Review and Comment	20 days	Wed 12/14/16	Tue 1/10/17	142
144	Conference Call to discuss USEPA Comments	1 day	Tue 1/17/17	Tue 1/17/17	143FS+4 days
145	Submit Final Screening Level Risk Assessment Report	15 days	Wed 1/11/17	Tue 1/31/17	143



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[1] - As required by the Dispute Resolution, dated December 15, 2010
[2] - Corresponds to Phase 2A or Phase 2B, detailed in the Final Work Plan for Operable Unit One (OU1) Groundwater and Data Gap Investigation - Phase 1A (Work Plan), dated May 10, 2013

QAPP Worksheet #17 - Sampling Design and Rationale

Please refer to Steps 4 and 7 in the DQO tables, contained in Worksheets #11-1 through #11-6.

QAPP Worksheet #18 - Sampling Locations and Methods/SOP Requirements

SAMPLING LOCATION/ID NUMBER	*MATRIX*	*ANALYTICAL GROUP*	*CONCENTRATION LEVEL*	*NUMBER OF SAMPLES (identify field duplicates)*	*SAMPLING SOP REFERENCE*	*RATIONALE FOR SAMPLING LOCATIONS*
Sampling location number to be specified prior to field work. Sample ID number to be specified during field work.	Water	VOCs	Low	Please refer to Steps 4 and 7 in the DQO tables, contained in Worksheets #11-1 through #11-6.	See Worksheet #21	Please refer to Steps 4 and 7 in the DQO tables, contained in Worksheets #11-1 through #11-6 and *Operable Unit Two (OU2) Remedial Investigation/Feasibility Study (RI/FS) Work Plan (CRA, 2014)*
	Water	SVOCs	Standard		See Worksheet #21	
	Water	PCBs	All		See Worksheet #21	
	Water	Pesticides	All		See Worksheet #21	
	Water	Herbicides	All		See Worksheet #21	
	Water	Dissolved Gases	N/A		See Worksheet #21	
	Water	Metals	All		See Worksheet #21	
	Water	Mercury	All		See Worksheet #21	
	Water	Cyanide	All, total		See Worksheet #21	
	Water	Alkalinity	All		See Worksheet #21	
	Water	Anions	All		See Worksheet #21	
	Water	TOC	All		See Worksheet #21	
	Water	pH/ Corrosivity	N/A		See Worksheet #21	
	Water	Sulfide	All		See Worksheet #21	
	Water	Hardness	All		See Worksheet #21	
	Water	Flashpoint	N/A		See Worksheet #21	
	Water	Dioxins& Furans	All		See Worksheet #21	
Sampling location number to be specified prior to field work. Sample ID number to be specified during	Soil	VOCs	Low	Please refer to Steps 4 and 7 in the DQO tables, contained in Worksheets #11-1 through #11-6.	See Worksheet #21	Please refer to Steps 4 and 7 in the DQO tables, contained in Worksheets #11-1 through #11-6 and *Operable Unit Two*
	Soil	SVOCs	Standard		See Worksheet #21	
	Soil	PCBs	All		See Worksheet #21	
	Soil	Pesticides	All		See Worksheet #21	
	Soil	Herbicides	All		See Worksheet #21	

SAMPLING LOCATION/ID NUMBER	*MATRIX*	*ANALYTICAL GROUP*	*CONCENTRATION LEVEL*	*NUMBER OF SAMPLES (identify field duplicates)*	*SAMPLING SOP REFERENCE*	*RATIONALE FOR SAMPLING LOCATIONS*
field work.	Soil	ICP Metals	All		See Worksheet #21	*(OU2) Remedial Investigation/Feasibility Study (RI/FS) Work Plan (CRA, 2014)*
	Soil	Mercury	All		See Worksheet #21	
	Soil	Cyanide	All, total		See Worksheet #21	
	Soil	Total Solids, %Moisture	N/A		See Worksheet #21	
	Soil	Anions			See Worksheet #21	
	Soil	TOC			See Worksheet #21	
	Soil	pH/ Corrosivity	N/A		See Worksheet #21	
	Soil	Sulfide	All		See Worksheet #21	
	Soil	Flashpoint	N/A		See Worksheet #21	
	Soil	Dioxins& Furans	All		See Worksheet #21	
Sampling location number to be specified prior to field work. Sample ID number to be specified during field work.	Air	VOCs in air	All	Please refer to Steps 4 and 7 in the DQO tables, contained in Worksheets #11-1 through #11-6.	See Worksheet #21	Please refer to Steps 4 and 7 in the DQO tables, contained in Worksheets #11-1 through #11-6 and *Operable Unit Two (OU2) Remedial Investigation/Feasibility Study (RI/FS) Work Plan (CRA, 2014)*

QAPP Worksheet #19 - Analytical SOP Requirements

ANALYTE/ANALYTE GROUP	*MATRIX*	*METHOD/ SOP*	*ACCREDITATION EXPIRATION DATE*	*CONTAINER(S) (number, size & type per sample)*	*PRESERVATION*	*PREPARATION HOLDING TIME*	*ANALYTICAL HOLDING TIME*
Volatile Organic Compounds	Water	SW846 5030, 8260 / NC-MS-019		3 x 40-mL VOA vials	pH ≤ 2 with HCl Cool to 4 ± 2°C	N/A	14 days
Semi Volatile Organic Compounds	Water	SW846 3520, 8270 / NC-OP-037 NC-MS-018		1 x 1-L Amber Glass Bottle	Cool to 4 ± 2°C	7 days	40 days
PCB	Water	SW846 3520C, 8082 / NC-OP-037, NC-GC-038		1 x 1-L Amber Glass Bottles	Cool to 4 ± 2 °C	7 days	40 days
Pesticides	Water	SW846 3520C 8081A / NC-GC-038, NC-OP-037		1 x 1-L Amber Glass Bottles	Cool to 4 ± 2 °C	7 Days	40 Days
Herbicides	Water	SW846 8151A / NC-OP-031 NC-GC-038		1 x 1-L Amber Glass Bottles	Cool to 4 ± 2 °C	7 Days	40 Days
Dissolved Gases	Water	Method RSK-175 / NC-GC-032		3 x 40-mL Vials	pH ≤ 2 with HCl Cool to 4 ± 2 °C		14 days

ANALYTE/ANALYTE GROUP	MATRIX	METHOD/ SOP	ACCREDITATION EXPIRATION DATE	CONTAINER(S) (number, size & type per sample)	PRESERVATION	PREPARATION HOLDING TIME	ANALYTICAL HOLDING TIME
ICP Metals	Water	EPA 200.7, SW846 6010 and 7000 series / NC-MT-012; SW846 3005A and 3010A / NC-IP-011		1 x 100-mL HDPE	HNO3 to pH $\leq$2, Cool to 4 $\pm$ 2 °C		180 Days
Mercury	Water	MCAWW 245.1, SW846 7470 / NC-MT-014		1 x 100-mL HDPE	HNO3 to pH $\leq$2		28 Days
Cyanide	Water	SW-846 9012A, EPA 335.2, 335.4, and SM 4500CN-E, 4500CN-G, 4500CN-I / NC-WC-031, NC-WC-032		1 x 500-mL plastic or glass	4 $\pm$2 °C, NaOH to pH>12		14 Days
Alkalinity	Water	EPA 310.1, SM 2320B / NC-WC-093	18 July 2016	1 x 500-mL Plastic	Cool to 4 + 2 °C		14 Days
Anions	Water	EPA 300.0, SW846 9056 / NC-WC-084		1 x 100-mL Plastic	Cool to 4 + 2 °C		28 Days or 48 Hours**
Total Organic Carbon	Water	EPA 415.1, SW846 9060A, SM5310C / NC-WC-017		3 x 40-mL G-TLS	H2SO4 to pH $\leq$2, Cool to 4 $\pm$ 2 °C		28 Days

ANALYTE/ANALYTE GROUP	MATRIX	METHOD/ SOP	ACCREDITATION EXPIRATION DATE	CONTAINER(S) (number, size & type per sample)	PRESERVATION	PREPARATION HOLDING TIME	ANALYTICAL HOLDING TIME
pH / Corrosivity	Water	EPA 150.1, SM 4500H+B, SW846 9040 / NC-WC-010		1 x 100-mL Plastic	Cool to 4 ± 2 °C		24 Hours*
Sulfide	Water	SW846 9030B, 9034, EPA 376.1, SM 4500S2-E / NC-WC-060		1 x 500-mL Plastic	Zn Acetate, NaOH to pH≥9, Cool to 4 ± 2 °C		7 Days
Hardness	Water	EPA 130.2, SM 2340C / NC-WC-036		1 x 250-mL Plastic or glass	Preserve to pH <2 with Nitric Acid Cool to 4 ± 2 °C		6 months
Flashpoint	Water	SW846 1010, ASTM D93-08 / NC-WC-034		1 x 1000-mL glass	Cool to 4 ± 2 °C		28 Days
Dioxins &Furans	Water	SW846 8290, 8290A, TO-9A / WS-ID-0005 WS-IDP-0005		1 x 1-L Amber Glass Bottles	Cool to 4 ± 2 C	30 days for extraction	45 days for analysis
Volatile Organic Compounds	Solid	SW846 5035, 8260C / NC-MS-019		3-EnCore® devices or equivalent	4 ± 2°C	48 hours to prep or freeze	14 days
Semi Volatile Organic Compounds	Solid	SW846 3540, 8270 / NC-OP-040 NC-MS-018		1 x 8-ounce glass jar with Teflon®-lined lid	Cool to 4 ± 2°C	14 days	40 days

ANALYTE/ANALYTE GROUP	MATRIX	METHOD/ SOP	ACCREDITATION EXPIRATION DATE	CONTAINER(S) (number, size & type per sample)	PRESERVATION	PREPARATION HOLDING TIME	ANALYTICAL HOLDING TIME
PCB	Solid	SW846 3540C, 8082 / NC-OP-040, NC-GC-038		1 x 8-ounce glass jar with Teflon®-lined lid or stainless steel liner	Cool to 4 ± 2 °C	14 days	40 days
Pesticides	Solid	SW846 3540C, 8081A / NC-GC-038 NC-OP-040		1 x 8-ounce glass jar with Teflon®-lined lid or stainless steel liner	Cool to 4 ± 2 °C	14 Days	40 Days
Herbicides	Solid	SW846 8151A / NC-OP-031 NC-GC-038		1 x 8-ounce glass jar with Teflon®-lined lid or stainless steel liner	Cool to 4 ± 2 °C	14 Days	40 Days
ICP Metals	Solid	SW846 6010, 7000 series / NC-MT-012; Method 3050B / NC-IP-010		1 x 8-ounce glass jar with Teflon®-lined lid or stainless steel liner	Cool to 4 ± 2 °C		180 Days
Mercury	Solid	SW846 7471 / NC-MT-014		1 x 8-ounce glass jar with Teflon®-lined lid or stainless steel liner	Cool to 4 ± 2 °C		28 Days
Cyanide	Solid	SW846 9012A, EPA 335.2, 335.4, SM 4500CN-E, 4500CN-G, 4500CN-I / NC-WC-031 NC-WC-032		1 x 8-ounce glass jar with Teflon®-lined lid or stainless steel liner	Cool to 4 ±2 °C		14 Days

ANALYTE/ANALYTE GROUP	MATRIX	METHOD/ SOP	ACCREDITATION EXPIRATION DATE	CONTAINER(S) (number, size & type per sample)	PRESERVATION	PREPARATION HOLDING TIME	ANALYTICAL HOLDING TIME
Total Solids. Percent Moisture, Total Settleable Solids	Solid	EPA 160.3, 160.5, ASTM D2216-98 / NC-WC-004		Glass or plastic containers	Cool to 4 ± 2 °C		N/A
Anions	Solid	EPA 300.0, SW846 9056 / NC-WC-084		1 x 8-ounce glass jar with Teflon®-lined lid or stainless steel liner	Cool to 4 + 2 °C		28 Days
Total Organic Carbon	Solid	Walkley Black NC-WC-018		1 x 8-ounce glass jar with Teflon®-lined lid or stainless steel liner	Cool to 4 ± 2 °C		28 Days
pH / Corrosivity	Solid	SW 846 9045 / NC-WC-010		1 x 8-ounce glass jar with Teflon®-lined lid	Cool to 4 ± 2 °C		24 Hours*
Sulfide	Solid	SW846 9030B, 9034 / NC-WC-060		1 x 8-ounce glass jar with Teflon®-lined lid	Cool to 4 ± 2 °C		7 Days
Flashpoint	Solid	SW846 1010, ASTM D93-08 / NC-WC-034		1 x 16-ounce glass jar with Teflon®-lined lid	Cool to 4 ± 2 °C		28 Days
Dioxins &Furans	Solid	SW846 8290, 8290A, TO-9A / WS-ID-0005 WS-IDP-0005		1 x 8-ounce glass jar with Teflon®-lined lid or stainless steel liner	Cool to 4 ± 2 °C	30 days for extraction	45 days for analysis

ANALYTE/ANALYTE GROUP	MATRIX	METHOD/ SOP	ACCREDITATION EXPIRATION DATE	CONTAINER(S) (number, size & type per sample)	PRESERVATION	PREPARATION HOLDING TIME	ANALYTICAL HOLDING TIME
Volatile Organic Compounds	Air	TO-15 / KNOX-MS-0001	30 June 2014	6-liter Summa canister, 1-liter Summa canister		N/A	30 days

**Samples should be analyzed as soon as possible after sampling, but not to exceed one day after sampling.*

** Anions Nitrite, Nitrate, and Ortho Phosphate have a maximum holding time of 48 hours from sampling.

QAPP Worksheet #20 - Field Quality Control Sample Summary

MATRIX	*ANALTICAL GROUP*	*CONCENTRATION LEVEL*	*ANALYTICAL AND PREPARATION SOP REFERENCE[(1)]*	*NUMBER OF SAMPLING LOCATIONS*	*NUMBER OF FIELD DUPLICATE PAIRS*	*MATRIX SPIKES*	*NUMBER OF TRIP BLANKS*	*FIELD BLANKS*	*NUMBER OF EQUIPMENT BLANKS*	*TOTAL NUMBER OF SAMPLES TO LABORATORY*
Water	VOCs	Low	D-1	TBD	1 per 10 samples	1 / 20	1 per cooler	1 / day	1 / day	TBD
Water	SVOCs	Standard	D-2, E-1	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD
Water	PCBs	All	D-3, E-1	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD
Water	Pesticides	All	D-3, E-1	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD
Water	Herbicides	All	D-3, E-7	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD
Water	Dissolved Gases	N/A	D-4	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD
Water	Metals	All	D-5, E-9	TBD	1 per 10 samples	1 /20	N/A	1 / day	1 / day	TBD
Water	Mercury	All	D-6	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD
Water	Cyanide	All, total	D-7, E-5	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD
Water	Alkalinity	All	D-9	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD
Water	Anions	All	D-10	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD
Water	TOC	All	D-11	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD
Water	pH/ Corrosivity	N/A	D-12	TBD	1 per 10 samples		N/A	1 / day	1 / day	TBD
Water	Sulfide	All	D-13	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD
Water	Hardness	All	D-14	TBD	1 per 10 samples	1/20	N/A	1 / day	1 / day	TBD

MATRIX	*ANALTICAL GROUP*	*CONCENTRATION LEVEL*	*ANALYTICAL AND PREPARATION SOP REFERENCE[(1)]*	*NUMBER OF SAMPLING LOCATIONS*	*NUMBER OF FIELD DUPLICATE PAIRS*	*MATRIX SPIKES*	*NUMBER OF TRIP BLANKS*	*FIELD BLANKS*	*NUMBER OF EQUIPMENT BLANKS*	*TOTAL NUMBER OF SAMPLES TO LABORATORY*
Water	Flashpoint	N/A	D-15	TBD	1 per 10 samples		N/A	1 / day	1 / day	TBD
Water	Dioxins& Furans	All	D-16, E-6	TBD	1 per 10 samples	1 / 20	N/ A	1 / day	1 / day	TBD
Soil	VOCs	Low	D-1	TBD	1 per 10 samples	1 /20	1 per cooler	1 / day	1 / day	TBD
Soil	SVOCs	Standard	D-2, E-4	TBD	1 per 10 samples	1 /20	N/A	1 / day	1 / day	TBD
Soil	PCBs	All	D-3, E-4	TBD				1 / day	1 / day	TBD
Soil	Pesticides	All	D-3, E-4	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD
Soil	Herbicides	All	D-3, E-7	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD
Soil	ICP Metals	All	D-5, E-8	TBD	1 per 10 samples	1 /20	N/A	1 / day	1 / day	TBD
Soil	Mercury	All	D-6	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD
Soil	Cyanide	All, total	D-7, E-5	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD
Soil	Total Solids, %Moisture	N/A	D-8	TBD			N/A	1 / day	1 / day	TBD
Soil	Anions		D-10	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD
Soil	TOC		D-18	TBD				1 / day	1 / day	TBD
Soil	pH/ Corrosivity	N/A	D-12	TBD	1 per 10 samples		N/A	1 / day	1 / day	TBD
Soil	Sulfide	All	D-13	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD

MATRIX	*ANALTICAL GROUP*	*CONCENTRATION LEVEL*	*ANALYTICAL AND PREPARATION SOP REFERENCE*[(1)]	*NUMBER OF SAMPLING LOCATIONS*	*NUMBER OF FIELD DUPLICATE PAIRS*	*MATRIX SPIKES*	*NUMBER OF TRIP BLANKS*	*FIELD BLANKS*	*NUMBER OF EQUIPMENT BLANKS*	*TOTAL NUMBER OF SAMPLES TO LABORATORY*
Soil	Flashpoint	N/A	D-15	TBD	1 per 10 samples		N/A	1 / day	1 / day	TBD
Soil	Dioxins& Furans	All	D-16,E-6	TBD	1 per 10 samples	1 / 20	N/A	1 / day	1 / day	TBD
Air	VOCs	All	D-17	TBD	1 per 10 samples	N/A	1 per cooler	1 / day	1 / day	TBD

TBD = to be determined

N/A = not applicable

(1) See Analytical SOP Reference Sheet (Worksheet #23)

QAPP Worksheet #21 - Project Sampling SOP Reference

REFERENCE NUMBER	*TITLE, REVISION DATE, AND/OR NUMBER*	*ORIGINATING ORGANIZATION*	*EQUIPMENT TYPE*	*MODIFIED FOR PROJECT WORK? (Y/N)*	*COMMENTS*
FSP, Appendix J-F-15	Groundwater Sampling	CRA	pH meter, conductivity meter, ORP meter, nephelometer, DO meter; field filtration units; purging/sampling equipment (pumps, bailers), water level probe; sampling materials (containers, COC, coolers, forms); HASP		
G-1	Surface Water Sample Collection	CRA	pH meter, conductivity meter, ORP meter, nephelometer, DO meter; sampling materials (containers, COC, coolers, forms,); sampling equipment (pumps, bailers, pole, nylon rope), HASP; Watercraft as needed		
FSP, Appendix J-F-31	Test Pit and Trench Soil Sample Collection	CRA	Field Screening (PID, LEL meter); sampling equipment (trowel, shovel or stainless steel spoon, stainless steel bowl); sample materials (plastic bags, mason jars, coolers, forms, camera, COC, log books); HASP		

REFERENCE NUMBER	*TITLE, REVISION DATE, AND/OR NUMBER*	*ORIGINATING ORGANIZATION*	*EQUIPMENT TYPE*	*MODIFIED FOR PROJECT WORK? (Y/N)*	*COMMENTS*
FSP, Appendix J-F-32	Surficial Soil Sample Collection	CRA	Field Screening (PID, LEL meter); sampling equipment (trowel, shovel or stainless steel spoon, stainless steel bowl); sample materials (plastic bags, mason jars, coolers, forms, camera, COC, log books); HASP		
FSP, Appendix J-F-38	Sub-surface Soil Sampling with Geoprobe®	CRA	Field Screening (PID, Sudan IV); sampling equipment (En Core® Soil VOC sampler, stainless steel spoon, stainless steel bowl); sample materials (plastic bags, mason jars, Teflon lined vials, coolers, forms, camera, COC, log books); HASP		
FSP, Appendix J-F-34	Vertical Aquifer Sampling by Geoprobe®	CRA	pH meter, conductivity meter, ORP meter, nephelometer; field screening (PID, Sudan IV); sample materials (peristaltic pump, tubing, laboratory supplied analyte specific sample containers with necessary preservatives, coolers, forms, COC, log books); HASP		

REFERENCE NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	ORIGINATING ORGANIZATION	EQUIPMENT TYPE	MODIFIED FOR PROJECT WORK? (Y/N)	COMMENTS
FSP, Appendix J-F-38	Vertical Aquifer Sampling / Temporary Monitoring Well Installation and Sampling by Geoprobe®	CRA	pH meter, conductivity meter, ORP meter, nephelometer; field screening (PID, Sudan IV); sample materials (peristaltic pump, tubing, laboratory supplied analyte specific sample containers with necessary preservatives, coolers, forms, COC, log books); HASP		
FSP, Appendix J-F-13	Rotosonic Drilling Method (Vertical Aquifer Sampling and Monitoring Well Installation)	CRA	pH meter, conductivity meter, ORP meter, nephelometer; field screening (PID, Sudan IV); sample materials (submersible pump, tubing, laboratory supplied analyte specific sample containers with necessary preservatives, coolers, forms, COC, log books); HASP		
FSP, Appendix J-F-7	Hollowstem Leadslot Auger Borehole Advancement and Sample Collection	CRA	pH meter, conductivity meter, ORP meter, nephelometer; field screening (PID, Sudan IV); sample materials (submersible pump and packer, tubing, laboratory supplied analyte specific sample containers with necessary preservatives, containers or jars, coolers, forms, COC, log books); HASP		

REFERENCE NUMBER	*TITLE, REVISION DATE, AND/OR NUMBER*	*ORIGINATING ORGANIZATION*	*EQUIPMENT TYPE*	*MODIFIED FOR PROJECT WORK? (Y/N)*	*COMMENTS*
FSP, Appendix J-F-35	Composite Waste Type Sampling	CRA	Field screening (PID, LEL meter); sampling equipment (trowel, shovel or stainless steel spoon, stainless steel bowl); sample materials (plastic bags, mason jars, coolers, forms, camera, COC, log books); HASP		
G-2	Sediment Sampling	CRA	Field Screening (PID, LEL meter,) sampling equipment (Piston Corer, stainless steel hand auger, stainless steel bowl, stainless steel spoon, jig or reciprocating saw); sample materials (plastic bags, mason jars, coolers, forms, camera, COC, log books); HASP		
FSP, Appendix J-F-11	Soil Gas Probe Sampling	CRA	PID (APRs if needed); Summa™ canisters, Teflon tubing, vacuum gauge, personal sampling pump, air tight stainless steel or brass tee-connectors and tee-valves; soil gas probe leak test material,; sample materials (forms, camera, COC, log books); HASP		
FSP, Appendix J-F-33	Gas Probe Installation	CRA	See Appendix J – F (Report 38443-7)		
FSP, Appendix J-F-37	Indoor Air Sampling	CRA	PID, Summa™ canisters, COC, field books, forms		

REFERENCE NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	ORIGINATING ORGANIZATION	EQUIPMENT TYPE	MODIFIED FOR PROJECT WORK? (Y/N)	COMMENTS
FSP, Appendix J-F-10	Landfill Gas Monitoring	CRA	Portable LFG analyzer (e.g. LandTec GA-90 /Gem500/GEM2000); oxygen meter, magnehelic pressure gage, digital manometer, liquid manometer, electronic water level indicator, pitot tube, digital thermometer; additional portable combustible meters (catalytic oxidation detectors. thermal conductivity detector, infrared gas analyzer, solid state sensor, electrochemical sensor,); flow meter, portable air monitor, water level meter		
FSP, Appendix J-F-19	pH/Temperature Measurement	CRA	Temperature compensated pH meter, YSI Model 3560 Water Quality Monitoring System; Combination pH electrode YSI Model 3530; Thermilinear thermister YSI Model 3510 temperature probe; pH buffer solutions; distilled or Di water		
FSP, Appendix J-F-20	Oxidation/Reduction Potential (ORP) Measurement	CRA	ORP meter, YSI Model 3560 Water Quality Monitor; ORP electrode assembly YSI Model 3540; Thermilinear thermistor temperature probe YSI Model 3510; ZoBell Solution, YSI Model 3682; distilled or Di water		

REFERENCE NUMBER	*TITLE, REVISION DATE, AND/OR NUMBER*	*ORIGINATING ORGANIZATION*	*EQUIPMENT TYPE*	*MODIFIED FOR PROJECT WORK? (Y/N)*	*COMMENTS*
FSP, Appendix J-F-21	Conductivity Measurement	CRA	Conductivity meter - YSI Model 3560 Water Quality Monitoring System; Conductivity Cell - YSI Model 3520 Flow-Through Conductivity Cell (K=5/cm); Thermilinear Thermister - YSI Model 3510 Temperature Probe; Conductivity standard, 1.0 mmhos/cm @25°C - YSI Model 3167; DI water		
FSP, Appendix J-F-22	Dissolved Oxygen Measurement	CRA	Temperature compensated dissolved oxygen (DO) meter, YSI Model 52; DO probe, YSI 5739 Field Probe; DO probe electrolyte solution; DO membrane replacement kit; Distilled water		
FSP, Appendix J-F-23	Turbidity	CRA	Direct reading turbidity meter, HF Scientific Model DRT-15C; Cuvettes with screw tops; Battery charger; 0.02 NTU (nominal) reference standard; distilled or DI water		
FSP, Appendix J-F-9	Soil VOC Screening	CRA	Mini Rae Plus Classic Photoionization Detector (PID) 10.6 eV and 11.7 eV lamp choice; calibrations gas; calibration apparatus and tubing; battery chargers		

REFERENCE NUMBER	*TITLE, REVISION DATE, AND/OR NUMBER*	*ORIGINATING ORGANIZATION*	*EQUIPMENT TYPE*	*MODIFIED FOR PROJECT WORK? (Y/N)*	*COMMENTS*
FSP, Appendix J-F-16	Field Filtering	CRA	In-line disposable 0.45 µm filter cartridges, sample tubing, pump, sample vials		
FSP, Appendix J-F-24	En Core® Soil VOC Sampler	CRA	Disposable En Core® sampler		
FSP, Appendix J-F-28	Field Screening of NAPL	CRA	SUDAN IV Test kit or approved equivalent		
FSP, Appendix J-F-26	Photoionization / Flame Ionization Detectors	CRA	Foxboro TVA - 1000B Toxic Vapor Analyzer; calibration gas; calibration apparatus and tubing; battery charger		
FSP, Appendix J-F-27	Non-Aqueous Phase Liquid (NAPL) and Water Level Monitoring	CRA	Dual phase oil/water interface probe; Solinst™ electric water level tape (or equivalent)		
FSP, Appendix J-F-25	Field Log Books and Photo Logs	CRA	Bound log book; high quality camera, dry erase board, dry erase marker		
FSP, Appendix J-F-1	Global Positioning System (GPS) Unit Operation	CRA	Trimble GeoXH handheld (accuracy to 10 centimeters) or equivalent; Leica GS50 (accuracy to 50 centimeters) or equivalent; Garmin eTrex Legend HCx handheld (accuracy to 3 meters) or equivalent		
FSP, Appendix J-F-29	Radiation Monitoring	CRA	Low level radiation meter (e.g., Victoreen Survey Meter)		

FSP – Field Sampling Plan 038443-Rpt 07 (CRA, 2013)

QAPP Worksheet #22 - Field Equipment Calibration, Maintenance, Testing, and Inspection

FIELD EQUIPMENT	*CALIBRATION ACTIVITY*	*MAINTENANCE ACTIVITY*	*TESTING ACTIVITY*	*INSPECTION ACTIVITY*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*REPSONSIBLE PERSON*	*SOP REFERENCE*[1]
Photoionization Detector	FSP, Appendix J-F -26	Per manufacturer's specifications	Measuring photoionization in soil	Check sample device	Daily	FSP, Appendix J-F -26			FSP, Appendix J-F -26
Water Level Meter	FSP, Appendix J-F -27	Per manufacturer's specifications	Water levels	Check sample device	Daily	FSP, Appendix J-F -27			FSP, Appendix J-F -27
Low Level Radiation Meter (Victoreen Survey Meter)	Factory Calibrated	Per manufacturer's specifications	Measuring ionizing radiation	Check sample device	Daily	N/A			FSP, Appendix J-F -29

[1] See Project Sampling SOP Reference table (Worksheet #21)

QAPP Worksheet #23 - Analytical SOP References

23-1 - Laboratory Sample Analysis

REFERENCE NUMBER	*SOP #*	*TITLE, DATE, AND URL (if available)*	*DEFINITIVE OR SCREENING DATA*	*MATRIX/ANALYTICAL GROUP*	*INSTURMENT/ EQUIPMENT TYPE*	*MODIFIED FOR PROJECT WORK? (Y/N)*	*ORGANIZATION PERFORMING ANALYSIS*
D-1	NC-MS-019	Determination of Volatile Organics by GC/MS 8260A, 8260B, and 8260C, Rev. 3-A Effective 6/29/12	Definitive	Water and Solids/MSV / VOCs	Gas Chromatography/ Mass Spectroscopy (GC/MS)		TA – NC
D-2	NC-MS-018	GC/MS Analysis Based on Methods 8270C and 8270D, Revision 3, Effective 4/25/13	Definitive	Water and Solid/MSS / SVOCs	(GC/MS)		TA – NC
D-3	NC-GC-038	Gas Chromatographic Analysis Based on Methods 8000B, 8081A, 8081B, 8082, 8082A, 8151A, 8015B, and 8015C Rev 3, Effective 4/18/13	Definitive	Water and Solid / PCBs, Pesticides, Herbicides	Gas Chromatography/ Electron Capture Detector (GC-ECD)		TA – NC
D-4	NC-GC-032	Analysis of Dissolved Gases in Groundwater by Modified Method RSK-175, Rev 5, Effective 4/29/13	Definitive	Water/ Dissolved Gases	N/A		TA – NC
D-5	NC-MT-012	Inductively Coupled Plasma – Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis Methods 6010B, 6010C, and 200.7, Rev 4, Effective 9/13/13	Definitive	Solid and Water ICP / Metals	Inductively Coupled Plasma OES - Axial		TA – NC

REFERENCE NUMBER	*SOP #*	*TITLE, DATE, AND URL (if available)*	*DEFINITIVE OR SCREENING DATA*	*MATRIX/ANALYTICAL GROUP*	*INSTURMENT/ EQUIPMENT TYPE*	*MODIFIED FOR PROJECT WORK? (Y/N)*	*ORGANIZATION PERFORMING ANALYSIS*
D-6	NC-MT-014	Preparation and Analysis of Mercury in Aqueous and Solid Samples by Cold Vapor Atomic Absorption Spectroscopy Methods 245.1, 7470A, 7471A and 7471B; Rev 3, Effective 6/05/13	Definitive	Water and Solid / Mercury	Cold Vapor Atomic Absorption Spectroscopy (CVAA)		TA – NC
D-7	NC-WC-031	Cyanide Automated, Pyridine-Barbituric Acid Method [Method: SW846 902A, EPA Methods 335.2, 335.4, and Standard Methods 4500CN-E, 4500CN-I, and 4500CN-G] Rev 9, Effective Date: 3/22/13	Definitive	Water and solid / Cyanide	Konelab AquaChem		TA – NC
D-8	NC-WC-004	Total Solids, Percent Moisture, and Total Settleable Solids Method 160.3, EPA 160.5, ASTM D2216-98, Rev 3.5, Effective 4/23/12	Definitive	Solid/ Percent Solids, Percent Moisture	N/A		TA – NC
D-9	NC-WC-093	Total, Carbonate, Bicarbonate, and Hydroxide Alkalinity [Method: SM2320B and EPA 310.1] Rev 1, Effective Date: 9/19/13	Definitive	Water / Alkalinity	Autotitrator		TA – NC

REFERENCE NUMBER	*SOP #*	*TITLE, DATE, AND URL (if available)*	*DEFINITIVE OR SCREENING DATA*	*MATRIX/ANALYTICAL GROUP*	*INSTURMENT/ EQUIPMENT TYPE*	*MODIFIED FOR PROJECT WORK? (Y/N)*	*ORGANIZATION PERFORMING ANALYSIS*
D-10	NC-WC-084	Determination of Inorganic Anions by Ion Chromatography [Method: EPA Method 300.0A and SW-846 Method 9056A] Rev 12, Effective Date: 8/28/13	Definitive	Water and Solid / Anions	IC DX-120, IC DX-320, ICS2100		TA – NC
D-11	NC-WC-017	Total Organic Carbon (TOC) Method 9060, 415.1, and SM5310C, Rev 3, Effective 7/25/13	Definitive	Water/ TOC	OI Analytical 1010		TA – NC
D-12	NC-WC-010	pH Electrometric Method Methods 9040B, 9040C, 9041A, 9045C, 150.1, and SM4500 H+B Rev 12, Effective 03/21/13	Definitive	Water and Solid / pH (Corrosivity)	Orion Star A211		TA – NC
D-13	NC-WC-060	Sulfide Methods 9030B, 9034, 376.1, and SM4500S2-E Rev 8, Effective 08/12/13	Definitive	Water and Solid / Sulfide	N/A		TA – NC
D-14	NC-WC-036	Total Hardness (mg/L CaCo3), Titrimetric, EPA Method 130.2, SM 2340C, Rev 3.4, Effective 11/18/10	Definitive	Water/ Total Hardness	N/A		TA – NC
D-15	NC-WC-034	Flashpoint Closed Cup Method 1010 and ASTM D93-08, Rev 2, Effective 3/21/13	Definitive	Water and Solid / Flashpoint (Ignitability)	Herzog HFP-339		TA – NC

REFERENCE NUMBER	*SOP #*	*TITLE, DATE, AND URL (if available)*	*DEFINITIVE OR SCREENING DATA*	*MATRIX/ANALYTICAL GROUP*	*INSTURMENT/ EQUIPMENT TYPE*	*MODIFIED FOR PROJECT WORK? (Y/N)*	*ORGANIZATION PERFORMING ANALYSIS*
D-16	WS-ID-0005	Analysis of Samples for Polychlorinated Dioxins and Furans by HRGC/HRMS Methods 8290, 8290A, TO-9A, Rev 7.5, Effective 4/19/2013	Definitive	Water and Solid / Dixons & Furans	Gas Chromatography/ High-Resolution Spectrometry (GC/HRMS)		TA – NC
D-17	KNOX-MS-0001	VOA Canister Analysis, Revision 14, 7/16/13 (Based on EPA TO-14A, TO-15)	Definitive	Air/VOCs	Gas Chromatography/ Mass Spectroscopy (GC/MS)	N	TA – KX
D-18	NC-WC-018	Total Organic Carbon (TOC) Analysis for Non-Waters Method Walkley Black, Rev 3, Effective 5/10/13	Definitive	Solid /TOC	N/A		TA – NC

23-2 - Laboratory Sample Preparation

REFERENCE NUMBER	*SOP #*	*TITLE, DATE, AND URL (if available)*	*DEFINITIVE OR SCREENING DATA*	*MATRIX/ANALYTICAL GROUP*	*INSTURMENT OR EQUIPMENT TYPE*	*MODIFIED FOR PROJECT WORK? (Y/N)*	*ORGANIZATION PERFORMING ANALYSIS*
E-1	NC-OP-037	Continuous Liquid/Liquid Extraction of Organic Compounds from Waters Based on Method SW846 3520C and 600 Series and Waste Dilution Based on Method 3580A Rev 3, Effective 4/5/13	Definitive	Water Extraction /Pesticides	N/A		TA – NC
E-2	NC-OP-038	Separatory Funnel Extraction of Organic Compounds from Waters Based on Method SW846 3510C Revision 3 Effective: 5/14/13	Definitive	Water Extraction	N/A		TA – NC
E-3	NC-OP-039	Sonication Extraction of Organic compounds from Soils Based on Method SW846 3550C Rev. 1-A Effective 4/24/12	Definitive	Solid Extraction	N/A		TA – NC
E-4	NC-OP-040	Soxhlet (Traditional) Extraction of Organic Compounds From Soils Based on Method SW846 3540C and Waste Dilution Based on Method SW846 3580A (Rev. 1-a, Effective 4/24/12)	Definitive	Solid Extraction /Pesticides	N/A		TA – NC

REFERENCE NUMBER	*SOP #*	*TITLE, DATE, AND URL (if available)*	*DEFINITIVE OR SCREENING DATA*	*MATRIX/ANALYTICAL GROUP*	*INSTURMENT OR EQUIPMENT TYPE*	*MODIFIED FOR PROJECT WORK? (Y/N)*	*ORGANIZATION PERFORMING ANALYSIS*
E-5	NC-WC-032	Cyanide Preparation Method [Method: SW846 902A, EPA Methods 335.1, 335.2, 335.4, and Standard Methods 4500CN-E, 4500CN-I, and 4500CN-G] Rev 9, Effective Date 3/21/13	Definitive	Water and solid /Cyanide	N/A		TA – NC
E-6	WS-IDP-00 05	Preparation of Samples for Analysis of Polychlorinated Dioxins and Furans for Analysis HRGC/HRMS, Methods 8290, 8290A, TO-9A, Rev 7.5, Effective 4/19/13	Definitive	Water and solid/ Dioxins and Furans	N/A		TA – NC
E-7	NC-OP-031	Extraction Procedure for Chlorinated Acid Herbicides Based on Method 8151A [SW/846 Method 8151A] Rev 6, Effective 7/29/13	Definitive	Water and solid/ Herbicides	N/A		TA – NC
E-8	NC-IP-010	Acid Digestion for Solid Samples Method 3050B Rev 2, Effective 8/12/10	Definitive	Solid ICP/ Metals	N/A		TA – NC
E-9	NC-IP-011	Acid Digestion for Aqueous Samples Methods 3005A, 3010A and 200 Series, Rev 4, Effective 6/28/13	Definitive	Water ICP / Metals	N/A		TA – NC

REFERENCE NUMBER	*SOP #*	*TITLE, DATE, AND URL (if available)*	*DEFINITIVE OR SCREENING DATA*	*MATRIX/ANALYTICAL GROUP*	*INSTURMENT OR EQUIPMENT TYPE*	*MODIFIED FOR PROJECT WORK? (Y/N)*	*ORGANIZATION PERFORMING ANALYSIS*
E-10	NC-OP-033	Toxicity Characteristic Leaching Procedure and Synthetic Precipitation Leaching Procedure SW846 1311 and 1312, Rev 4, Effective 10/31/13	Definitive	Solids and Water / TCLP: VOCs, SVOCs, Pesticides, Herbicides, Metals, Ignitability, pH	N/A		TA – NC

23-3 - Laboratory Support

REFERENCE NUMBER	*SOP #*	*TITLE, DATE, AND URL (if available)*	*DEFINITIVE OR SCREENING DATA*	*MATRIX/ANALYTICAL GROUP*	*INSTURMENT OR EQUIPMENT TYPE*	*MODIFIED FOR PROJECT WORK? (Y/N)*	*ORGANIZATION PERFORMING ANALYSIS*
F-1	NC-QAM-001	Quality Assurance Manual, 5/23/12 Rev. 2A	N/A	All	N/A		TA – NC
F-2	QA-003	Quality Control Program, 10/31/13 Rev. 12	N/A	All	N/A		TA – NC
F-3	QA-020	Data Validation Response and Client Complaint Handling, 06/26/13 Rev. 8	N/A	All	N/A		TA – NC
F-4	NC-QA-012	Shipping Department, 9/13/13 Rev. 3	N/A		N/A		TA – NC
F-5	NC-QA-013	Inventory/Warehouse Control, 8/20/12 Rev. 1.7	N/A		N/A		TA – NC
F-6	NC-QA-014	Glassware Washing, 7/12/12 Rev. 10	N/A		N/A		TA – NC
F-7	NC-QA-018	Statistical Evaluation of Data and Development of Control Charts, 12/10/13 Rev. 14	N/A	Quality Control	N/A		TA – NC
F-8	NC-QA-021	Evaluation of Method Detection Limits for Chemical Tests, 9/30/13 Rev. 10	N/A	All	N/A		TA – NC
F-9	NC-QA-029	Nonconformance and Corrective Action System, 9/30/13 Rev. 4	N/A	All	N/A		TA – NC
F-10	NC-SC-005	Sample Receiving, 3/27/12 Rev. 6.9	N/A	Sample Management	N/A		TA – NC

QAPP Worksheet #24 - Analytical Instrument Calibration

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
GC/MS (VOA/8260B)	Check of mass spectral ion intensities (tuning procedure) using BFB (8260B)		Prior to ICAL and at the beginning of each 12-hour period.	Refer to method/SOP for specific ion criteria.	Retune instrument and verify.	Group Leader/ Analyst	NC-MS-019
GC/MS (VOA/8260B)	Minimum five-point initial calibration for target analytes, lowest concentration standard at or near the reporting limit. (ICAL)	Varies	Initial calibration prior to sample analysis	1) Average Response factor (RF) for SPCCs: VOCs > 0.30 for chlorobenzene and 1,1,2,2-PCA, > 0.10 for chloromethane, bromoform, and 1,1-dichloroethane 2) RSD for RFs for CCCs: <30% and one option below: a) RSD for each analyte <15%, b) linear least squares regression r > 0.995; c) Non-linear regression COD r-sq > 0.99, min 6 points for second order.	Evaluate standards, chromatography, and mass spectrometer response. If problem found with above, correct as appropriate, then repeat initial calibration.	Group Leader/Analyst	NC-MS-019
GC/MS (VOA/8260B)	Second-source calibration verification		Once after each ICAL	All project analytes within +20% of true value.	Evaluate data. If problem found, correct, then repeat second source verification. If it still	Group Leader/Analyst	NC-MS-019

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
					fails, then repeat initial calibration.		
GC/MS (VOA/8260B)	Retention Time Window Position Establishment		Once per ICAL, for each analyte and surrogate.	Set position using the mid-point standard of the ICAL when ICAL is performed. On days when ICAL is not performed, use initial CCV.	N/A	Group Leader/Analyst	NC-MS-019
GC/MS (VOA/8260B)	Evaluation of Relative Retention Times (RRT)		With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, rerun ICAL.	Group Leader/Analyst	NC-MS-019
GC/MS (VOA/8260B)	Daily calibration verification		Daily, prior to sample analysis and every 12 hours of analysis time.	1) Min RRF for SPCCs: RRF > 0.30 for chlorobenzene and 1,1,2,2-PCA, > 0.10 for chloromethane, bromoform, and 1,1-dichloroethane. 2) %Difference/ %Drift for all target compounds and surrogates: %D < 20%	Evaluate standard, chromatography, and mass spectrometer response. If problem found with above, correct as appropriate, then repeat CCV. If still fails, repeat initial calibration.	Group Leader/Analyst	NC-MS-019
GC/MS (VOA/8260B)	Internal Standards		Every field sample, standard, and QC sample.	Areas within -50% to +100% of last ICAL mid-point for each CCV.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed	Group Leader/Analyst	NC-MS-019

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
					while system was malfunctioning.		
GC/MS (SVOC/8270)	Check of mass spectral ion intensities (tuning procedure) using DFTPP (8270C)		Prior to ICAL and at the beginning of each 12-hour period.	Refer to method/SOP for specific ion criteria.	Retune instrument and verify.	Group Leader/Analyst	NC-MS-018
GC/MS (SVOC/8270)	Breakdown check		At the beginning of each 12-hour period, prior to analysis of samples.	Degradation < 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem then repeat breakdown check.	Group Leader/Analyst	NC-MS-018
GC/MS (SVOC/8270)	Minimum five-point initial calibration for target analytes, lowest concentration standard at or near the reporting limit. (ICAL)	Various	Initial calibration prior to sample analysis	1) Average Response factor (RF) for SPCCs: > 0.050 2) RSD for RFs for CCCs: <30% and one option below: a) RSD for each analyte <15%, b) linear least squares regression r > 0.995; c) non-linear regression COD r-sq > 0.99, min 6 points	Correct problem, then repeat initial calibration	Group Leader/Analyst	NC-MS-018

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
				for second order.			
GC/MS (SVOC/8270)	Second-source calibration verification		Once after each ICAL	All project analytes within +20% of true value.	Correct problem, and verify second source standard. Rerun verification. If still fails, repeat initial calibration.	Group Leader/Analyst	NC-MS-018
GC/MS (SVOC/8270)	Retention Time Window Position Establishment		Once per ICAL, for each analyte and surrogate.	Set position using the mid-point standard of the ICAL when ICAL is performed. On days when ICAL is not performed, use initial CCV.	N/A	Group Leader/Analyst	NC-MS-018
GC/MS (SVOC/8270)	Evaluation of Relative Retention Times (RRT)		With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, rerun ICAL.	Group Leader/Analyst	NC-QA-018
GC/MS (SVOC/8270)	Daily calibration verification (CCV)		Daily, prior to sample analysis and every 12 hours of analysis time.	1) Min RRF for SPCCs: >0.050 2) %Difference/ %Drift for all target compounds and surrogates: %D < 20%	Correct problem, then repeat. If still fails, repeat initial calibration. Reanalyze all samples since last successful calibration verification.	Group Leader/Analyst	NC-MS-018

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
GC/MS (SVOC/8270)	Internal Standards		Daily, prior to sample analysis and every 12 hours of analysis time.	Retention time + 30 seconds from retention time of the midpoint standard in the ICAL. Areas within -50% to +100% of last ICAL mid-point for each CCV.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Group Leader/Analyst	NC-MS-018
ICP Trace	ICAL	Various	Initial calibration prior to sample analysis	Correlation coefficient >0.995 (if more than one point); accepted if the initial calibration verification (ICV) passes	Evaluate standard and instrument response. If problem with instrument (autosampler failure, response poor, etc) or standards, correct as appropriate, then repeat initial calibration.	Group Leader / Analyst	NC-MT-012

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
ICP Trace	Low Concentration standard		Daily, after one point calibration	Within ± 20% of the true value for all target analytes	Evaluate standard and instrument response. If problem with instrument (autosampler failure, response poor, etc) or standards, correct as appropriate, then repeat initial calibration.	Group Leader / Analyst	NC-MT-012
ICP Trace	ICV (second source)		Once per initial calibration	Within ± 10% of the true value for all target analytes.	Evaluate standard and instrument response. If problem with instrument (autosampler failure, response poor, etc) or standards, correct as appropriate, then repeat initial calibration.	Group Leader / Analyst	NC-MT-012

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
ICP Trace	CCV		Following IC, after every 10 samples and the end of the sequence	Within ± 10% of the true value for all target analytes.	Evaluate standard and instrument response. If problem with instrument (autosampler failure, response poor, etc) or standards, correct as appropriate, then repeat. If still fails, repeat initial calibration. Re-analyze all samples since the last successful calibration verification.	Group Leader / Analyst	NC-MT-012
ICP Trace	ICB/CCB		After IC, after CCV calibration, after every 10 samples, and at the end of the sequence	No target analytes detected > RL.	Evaluate blank to determine if instrument or solution caused, then correct. Re-prepare and re-analyze the blank. All samples following the last acceptable calibration blank must be reanalyzed.	Group Leader / Analyst	NC-MT-012

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
ICP Trace	ICS		At the beginning of an analytical run	ICSA-A: Absolute values of concentration for all non-spiked analytes < RL (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ±20% of true value.	Terminate analysis, then reanalyze ICS and all affected samples.	Group Leader / Analyst	NC-MT-012
ICP Trace	LR (Linear Range)		Every six months for each analyte wavelength used for each instrument.	Within 10% of expected value.	Re-determine the LDR (Linear Dynamic Range).	Group Leader / Analyst	NC-MT-012
AquaChem	ICAL	0 to 0.20 mg/L	Initial calibration daily prior to sample analysis	Correlation coefficient >0.995; accepted if the initial calibration verification (ICV) passes	Evaluate standard and instrument response. If problem with instrument (autosampler failure, response poor, etc) or standards, correct as appropriate, then repeat initial calibration.	Group Leader / Analyst	NC-WC-031

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
AquaChem	ICV (second source)		Once per initial calibration	Less than 10% difference from IC for all target analytes	Evaluate standards and instrument response. If standard issue, repeat or remake then repeat standard as appropriate. If still fails, repeat initial calibration.	Group Leader / Analyst	NC-WC-031
AquaChem	CCV		Following IC, after every 10 samples and the end of the sequence	Less than 10% difference from IC for all target analytes	Evaluate standard and instrument response. If problem with instrument (autosampler failure, response poor, etc.) or standards, correct as appropriate, then repeat. If still fails, repeat initial calibration. Re-analyze all samples since the last successful calibration verification.	Group Leader / Analyst	NC-WC-031

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
AquaChem	ICB/CCB		Following ICV, after every 10 samples and the end of the sequence after each CCV	Results < RL	Change CCB solution, and check rinse solution. Evaluate standard and instrument response. If problem with instrument (autosampler failure, response poor, etc.) or standards, correct as appropriate, then repeat. If still fails, repeat initial calibration. Re-analyze all samples since the last successful calibration blank.	Group Leader / Analyst	NC-WC-031
CVAA	ICAL		Daily initial calibration prior to sample analysis	Correlation coefficient >0.995; accepted if the initial calibration verification (ICV) passes	Evaluate standard and instrument response. Repeat ICAL.	Group Leader / Analyst	NC-MT-014

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
CVAA	ICV		Once per each initial calibration, prior to beginning a sample run	Less than 10% difference from IC for all target analytes	Evaluate standards and instrument response. If standard issue, repeat or remake then repeat standard as appropriate. If still fails, repeat initial calibration.	Group Leader / Analyst	NC-MT-014
CVAA	CCV		Following IC, after every 10 samples and the end of the sequence	Less than 20% difference from IC	Evaluate standard and instrument response. If problem with instrument (autosampler failure, response poor, etc) or standards, correct as appropriate, then repeat. If still fails, repeat initial calibration. Re-analyze all samples since the last successful calibration verification.	Group Leader / Analyst	NC-MT-014

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
CVAA	CRA		At the beginning of each sample analysis run after the ICV/ICB pair.	TV + 50%	Standard is either rerun, or the problem corrected and the instrument recalibrated	Group Leader / Analyst	NC-MT-014
CVAA	ICB/CCB		Immediately following every CCV (ICV).	No result $\geq$ RL	Evaluate blank to determine if caused by instrument or solution, then correct. Re-prepare and re-analyze the blank. All samples following the last acceptable calibration blank must be reanalyzed.	Group Leader / Analyst	NC-MT-014
GC-ECD (Pesticides/8081A)	Breakdown Check		At the beginning of each 12-hour period, prior to analysis of samples.	Degradation $\leq$ 15% for both DDT and Endrin	Evaluate standard, chromatography, and detector response. If problem (e.g., active sites on column, dirty inlet) indicated, correct as appropriate, then repeat breakdown check.		NC-GC-038

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
GC-ECD (Pesticides/8081A)	Minimum five-point initial calibration (ICAL) for target analytes, lowest concentration standard at or near the reporting limit		Initial calibration prior to sample analysis	One of the options below: 1): RSD for each analyte ≤ 20%; 2) Linear least squares regression: r ≥ 0.995; 3) non-linear regression: COD (r2) ≥ 0.99, minimum of 6 points for second order.	Evaluate standards, chromatography, and detector response. If problem found with above, correct as appropriate, then repeat initial calibration		NC-GC-038
GC-ECD (Pesticides/8081A)	Retention Time Window Position Establishment		Once per ICAL, for each analyte and surrogate.	Set position using the mid-point standard of the ICAL when ICAL is performed. On days when ICAL is not performed, use initial CCV.	N/A		NC-GC-038
GC-ECD (Pesticides/8081A)	Second-source calibration verification (ICV)		Immediately following ICAL.	All project analytes within ± 20% of the expected value from the ICAL.	Evaluate data. If problem (e.g., concentrated standard, plugged injector needle) found, correct, then repeat second source verification. If still fails, repeat initial calibration.		NC-GC-038
GC-ECD (Pesticides/8081A)	Continuing calibration verification		Prior to sample analysis, after every 10 field	All project analytes within ± 20% of the expected value from	Evaluate standard, chromatography, and detector		NC-GC-038

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
	(CCV)		samples, and at the end of the sequence.	the ICAL.	response. If problem found with above, correct as appropriate, then repeat CCV. If still fails, repeat initial calibration. Re-analyze all samples since the last successful calibration verification.		
GC-ECD (Herbicides/ 8151A)	5-Pt ICAL		Initial calibration prior to sample analysis	One of the options below: 1): RSD for each analyte ≤ 20%; 2) Linear least squares regression: r ≥ 0.995; 3) non-linear regression: COD (r2) ≥ 0.99, minimum of 6 points for second order.	Evaluate standards, chromatography, and detector response. If problem found with above, correct as appropriate, then repeat initial calibration	Group Leader / Analyst	NC-GC-038

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
GC-ECD (Herbicides/ 8151A)	Retention Time Window Position Establishment		Once per ICAL and at the beginning of the analytical shift.	Set position using the mid-point standard of the ICAL when ICAL is performed. On days when ICAL is not performed, use initial CCV.	N/A	Group Leader / Analyst	NC-GC-038
GC-ECD (Herbicides/ 8151A)	Second-source calibration verification (ICV)		Immediately following ICAL.	All project analytes within ± 15% for TCL analytes and +30% for all other analytes of the expected value from the ICAL.	Evaluate data. If problem (e.g., concentrated standard, plugged injector needle) found, correct, then repeat second source verification. If still fails, repeat initial calibration.	Group Leader / Analyst	NC-GC-038
GC-ECD (Herbicides/ 8151A)	Continuing calibration verification		A CCV is analyzed after every 12 hours.	Retention time windows are updated with continuing calibration verifications.	N/A	Group Leader / Analyst	NC-GC-038
Autotitrator	Initial calibration of pH buffers 4, 7, and 10		Initial calibration daily prior to sample analysis.	pH7 buffer (ICV and CCV) = 7± .05	Refill pH probe solution, clean titration cell, recalibrate	Group Leader / Analyst	NC-WC-093

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
Autotitrator	pH7 (CCV)		Beginning and end of every sequence, and once every ten samples.	pH7 buffer = 7± .05	Refill pH probe solution, clean titration cell, recalibrate	Group Leader / Analyst	NC-WC-093
IC	ICAL	Various	Calibrate monthly, or more often as needed	Correlation coefficient >0.995 for linear; accepted if the initial calibration verification (ICV) passes, Average requires R^2 value < 20.	Evaluate standard and instrument response. If problem with instrument (e.g., autosampler failure, response poor, etc) or standards, correct as appropriate, then repeat initial calibration.	Group Leader / Analyst	NC-WC-084
IC	ICV		Once per initial calibration, immediately following ICAL, or at the beginning of each run.	Less than 10% difference from IC for all target analytes	Evaluate standards and instrument response. If standard issue, repeat or remake then repeat standard as appropriate. If still fails, repeat initial calibration.	Group Leader / Analyst	NC-WC-084

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
IC	CCV		After every 10 samples and the end of the sequence	Less than 10% difference from IC for all target analytes	Evaluate standard and instrument response. If problem with instrument (e.g., autosampler failure, response poor, etc.) or standards, correct as appropriate, then repeat. If still fails, repeat initial calibration. Re-analyze all samples since the last successful calibration verification.	Group Leader / Analyst	NC-WC-084
IC	ICB/CCB		Following ICV, every CCV, and end of run.	No target analyte $\geq$ RL for any anion.	Evaluate standard and instrument response. If problem with instrument (e.g., autosampler failure, response poor, etc.) or standards, correct as appropriate, then repeat. If still fails, repeat initial calibration. Re-analyze all samples since the last successful calibration blank.	Group Leader / Analyst	NC-WC-084

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
OI Analytical 1010	ICAL	1.0 to 50 mg/L	As needed	Correlation Coefficient $\geq$ 0.995, and ICV passes criteria	Evaluate standard and instrument response. If problem with instrument (e.g., autosampler failure, response poor, etc.) or standards, correct as appropriate, then repeat initial calibration.	Group Leader / Analyst	NC-WC-017
OI Analytical 1010	ICV/CCV		Once per initial calibration (ICV). CCVs at the beginning and end of each sequence, and every 10 samples.	$\pm$ 10% of the true value.	Evaluate standards and instrument response. If standard issue, repeat or remake then repeat standard as appropriate. If still fails, repeat initial calibration.	Group Leader / Analyst	NC-WC-017

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
OI Analytical 1010	ICB/CCB		Immediately following ICV (ICB) and CCV (CCB)	No analyte $\geq$ the RL	Evaluate standard and instrument response. If problem with instrument (e.g., autosampler failure, response poor, etc) or standards, correct as appropriate, then repeat. If still fails, repeat initial calibration. Re-analyze all samples since the last successful calibration verification.	Group Leader / Analyst	NC-WC-017
Orion Star A211	Initial calibration: 5 point calibration.	pH 2 to pH 12	Initial calibration daily prior to sample analysis	pH 7 buffer check and LCS meet criteria	Check pH probe, refresh calibration buffers, recalibrate	Group Leader / Analyst	NC-WC-010
Orion Star A211	Continuing calibration verification: pH 7 buffer		Beginning and end of every sequence, and every 10 samples.	pH 7 $\pm$ 0.05 SU	Check pH probe, refresh pH 7 buffer, recalibrate, and reanalyze all samples since last passing pH 7.	Group Leader / Analyst	NC-WC-010

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
HRGC/HRMS	Tune / Mass Resolution Check (PFK)		At the beginning and the end of each 12-hour period of analysis.	Resolving power ≥ 10,000 at m/z=304.9842 & m/z=380.9760 + 5ppm of expected mass. Lock-mass ion between lowest and highest masses for each descriptor and level of reference ≤ 10% full-scale deflection.	Retune instrument & verify. Assess data for impact if end resolution is less than 10,000 narrate or reinject as necessary.	Lab Manager / Analyst	WS-ID-0005
HRGC/HRMS	GC Column Performance Check (CPSM/WDM per method)		Prior to ICAL or calibration verification.	Peak separation between 2,3,7,8-TCDD and other TCDD isomers result in a valley of ≤ 25%; and identification of all first and last eluters of the eight homologue retention time windows and documentation by labeling (F/L) on the chromatogram; and absolute retention times for switching from one homologous series	1) Readjust windows. 2) Evaluate system. 3) Perform maintenance. 4) Reanalyze CPSM. 5) No corrective action is necessary if 2,3,7,8-TCDD is not detected and the % valley is greater than 25%.	Lab Manager / Analyst	WS-ID-0005

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
				to the next ≥ 10 seconds for all components of the mixture.			
HRGC/HRMS	Minimum five-point initial calibration for target analytes, lowest concentration standard at or near the reporting limit. (ICAL)		ICAL prior to sample analysis, as needed by the failure of calibration verification, and when a new lot is used as a standard source for calibration verification, internal standard or recovery standard solutions.	RSD ≤ 20% for response factors for 17 unlabelled isomers & labelled IS, and ion abundance ratios within limits specified in SOP; and S/N ≥ 10:1for target analytes.	Evaluate standard and instrument response. If problem with instrument (e.g., autosampler failure, response poor, etc) or standards, correct as appropriate, then repeat initial calibration.	Lab Manager / Analyst	WS-ID-0005
HRGC/HRMS	Second-source calibration verification		Immediately following ICAL.	All project analytes within ± 30% of the expected value from the ICAL.	Evaluate standards and instrument response. If standard issue, repeat or remake then repeat standard as appropriate. If still fails, repeat initial calibration	Lab Manager / Analyst	WS-ID-0005

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
HRGC/HRMS	Calibration Verification (CCV)		At the beginning of each 12-hour period, and at the end of each analytical sequence.	Ion abundance ratios in accordance with SOP; and RF (unlabelled standards) within ± 20%D of average RF from ICAL; and RF (labelled standards) within ± 30%D of average RF from ICAL.	Correct problem, repeat calibration verification. If fails, repeat ICAL and reanalyze all samples analyzed since last successful CCV. End of Run CCV: If RF (unlabelled standards) > ± 20%D and ≤ ± 25%D and/or RF (labelled standards) > ± 30%D and ≤ ± 35%D of the average RF from ICAL use mean RF from bracketing CCVs to quantitate impacted samples. If bracketing CCVs differ by more than 25% RPD (unlabelled) or 35% RPD (labelled), run a new ICAL within 2 hours, and requantitate samples. Otherwise, reanalyze samples with positive detections.	Lab Manager / Analyst	WS-ID-0005

INSTRUMENT	*CALIBRATION PROCEDURE*	*CALIBRATION RANGE*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION (CA)*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*SOP REFERENCE*
GC/MS (VOC/TO-15)	Mass scale calibration verification using BFB (tuning)	N/A	Verify tune every 24 hours	Ion abundance within method specified ranges as listed in SOP	Inspect system; correct problem; rerun BFB.	Analyst	KNOX-MS-0001, Revision 14
GC/MS (VOC/TO-15)	Initial Calibration (ICAL) (minimum 5 point calibration)	Varies by analyte – Refer to SOP	Prior to sample analysis, after major instrument changes and when continuing calibration criteria are not met.	ICAL %RSD $\leq$30% with $\leq$2 analytes $\leq$40%, or linear / quadratic curve r^2 $\geq$0.990.	Inspect system; correct problem; repeat ICAL.	Analyst	KNOX-MS-0001, Revision 14
GC/MS (VOC/TO-15)	Initial Calibration Verification (ICV	N/A	After Initial Calibration; prior to sample analysis	%D <35%.	Inspect system; correct problem; reanalyze ICV or repeat ICAL.	Analyst	KNOX-MS-0001, Revision 14
GC/MS (VOC/TO-15)	Continuing Calibration Verification (CCV)	N/A	At the beginning of each 24 hour shift.	CCV %D <30%. Allowance for >30% if the compound meet the LCS criteria	Inspect system; correct problem; repeat CCV. If still unacceptable, repeat ICAL.	Analyst	KNOX-MS-0001, Revision 14

QAPP Worksheet #25 - Analytical Instrument and Equipment Maintenance, Testing, and Inspection

INSTRUMENT/ EQUIPMENT	*MAINTENANCE ACTIVITY*	*TESTING ACTIVITY*	*INSPECTION ACTIVITY*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*REFERENCE*
GC/MS	Clean sources, maintain vacuum pumps	Tuning	Instrument performance and sensitivity	Service vacuum pumps twice per year, other maintenance as needed	Tune and CCV pass criteria	Recalibrate instrument	TestAmerica Analyst	NC-MS-019, NC-MS-018
GC/MS	Change septum, clean injection port, change or clip column, install new liner, change trap	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	Tune and CCV pass criteria	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument.	TestAmerica Analyst	NC-MS-019, NS-MS-018
ICP Trace	Replace disposables, flush lines, clean injector and torch	Intensity of 1PPM Manganese STD within criteria	Check connections	Daily or as needed	Intensity of 1PPM Manganese STD within criteria	Replace, investigate injector, reanalyze	Group Leader / Analyst	NC-MT-012
ICP Trace	Replace pump windings	Monitor ISTD counts for variation	Instrument performance and sensitivity	As needed	Monitor ISTD counts for variation	Replace windings, recalibrate and reanalyze	Group Leader / Analyst	NC-MT-012
AquaChem	Replace diluent, water blank analysis	System cleanliness check	Instrument performance and cleanliness	Instrument performance and cleanliness	CCV pass criteria	Clean reagent water container and repeat water blank analysis	TestAmerica Chemist	NC-WC-031

INSTRUMENT/ EQUIPMENT	*MAINTENANCE ACTIVITY*	*TESTING ACTIVITY*	*INSPECTION ACTIVITY*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*REFERENCE*
CVAA	Replace disposables, flush lines	Sensitivity	Instrument performance and sensitivity	As needed	CCV pass criteria	Recalibrate	TestAmerica Analyst	NC-MT-014
GC-ECD	Change septum, clean injection port, change or clip column, install new liner	Detector signals and chromatogram review	Instrument performance and sensitivity	As needed	CCV passes criteria	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	TestAmerica Chemist	NC-GC-038
Autotitrator	Clean titration cell, adjust sample amount, refresh/replace pH probe filling solution.	Cleanliness /functionality	Instrument performance and sensitivity	As Needed	pH 7 buffer reads 7 $\pm$ .05	Troubleshoot and recalibrate	TestAmerica Chemist	NC-WC-093
DX-120, DX-320, ICS2100	Replace columns	Retention Times	Instrument performance and sensitivity	As needed	ICV/CCV pass criteria	Pump eluent through system and recalibrate	TestAmerica Chemist	NC-WC-084
OI Analytical 1010	Leak Test	Testing for Leaks	Instrument performance	As needed	CCV passes criteria	Call for Service	Group Leader / Analyst	NC-WC-017
OI Analytical 1010	Reagent blanks	System cleanliness	Bias	As needed	CCV passes criteria	Rerun blanks, recalibrate	Group Leader / Analyst	NC-WC-017
Orion Star A211	Electrode maintenance (refilling and cleaning)	Sensitivity	Instrument performance and sensitivity	As needed	Calibration and pH 7 check meet criteria	Refill probe and allow it to sit in pH 7 buffer for 24 hours. Use new probe.	Analyst	NC-WC-010
Herzog HFP-339	Check igniter coil	Instrument	Instrument	As needed	p-Xylene	Repair or replace	TestAmerica	NC-WC-034

INSTRUMENT/ EQUIPMENT	*MAINTENANCE ACTIVITY*	*TESTING ACTIVITY*	*INSPECTION ACTIVITY*	*FREQUENCY*	*ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION RESPONSIBLE FOR CORRECTIVE ACTION*	*REFERENCE*
		performance	performance		passes criteria	igniter coil	Analyst	
GC/HRMS	Parameter Setup	Physical check	Physical check	Initially; prior to DCC	Correct Parameters	Reset if incorrect	TestAmerica Chemist	WS-ID-0005
GC/HRMS	Tune Check	Instrument Performance	Conformance to instrument tuning	Initially; prior to DCC	Compliance to ion abundance criteria	Correct the problem and repeat tune check	TestAmerica Chemist	WS-ID-0005
GCMS (TO-15)	Clean source, change traps, replace filaments; maintain vacuum pumps	Refer to Worksheet #24	Refer to Worksheet #24	Service vacuum pumps twice per year; other maintenance as needed	Refer to Worksheet #24	Refer to Worksheet #24	Analyst	KNOX-MS-0001, Rev 14

QAPP Worksheet #26 - Sample Handling System (TestAmerica LABORATORIES)

SAMPLE COLLECTION, LABELING, PACKAGING, AND SHIPMENT	
Sample Collection & Labeling, Chain of Custody Form completion (personnel/ organization):	Greg Lewis, Jeremy Teepen, Jason Close, CRA
Sample Packaging (personnel/ organization):	Greg Lewis, Jeremy Teepen, Jason Close, CRA
Coordination of Shipment (personnel/ organization):	Greg Lewis, Jeremy Teepen, Jason Close, CRA
Type of Shipment/ Carrier:	Overnight courier or direct laboratory delivery / pickup
SAMPLE RECEIPT AND ANALYSIS	
Sample Receipt, Inspection, & Log-in (personnel/organization):	Denise Heckler, TA –NC (or designee) and / or Jamie McKinney, TA – KX (or designee)
Sample Custody and Storage (personnel/organization):	Denise Heckler, TA –NC (or designee) and / or Jamie McKinney, TA – KX (or designee)
Sample Preparation (personnel/organization):	Denise Heckler, TA –NC (or designee) and / or Jamie McKinney, TA – KX (or designee)
Sample Determinative Analysis (personnel/organization):	Denise Heckler, TA –NC (or designee) and / or Jamie McKinney, TA – KX (or designee)
SAMPLE ARCHIVING	
Field Sample Storage (number of days from sample collection):	30 days from submittal of final report
Sample Extract/Digestate Storage (number of days from extraction/digestion):	60 days from submittal of final report
Biological Sample Storage (number of days from sample collection):	N/A
SAMPLE DISPOSAL	
Personnel/Organization:	Denise Heckler, TA-NC (or designee) and / or Jamie McKinney TA – KX (or designee)
Number of Days from Analysis:	60 days minimum from submittal of final report

QAPP Worksheet #27 - Sample Custody Requirements

FIELD SAMPLE CUSTODY PROCEDURES (sample collection, packaging, shipment, and delivery to laboratory):

The field sampler is personally responsible for the care and custody of the samples until they are transferred to the laboratory or properly dispatched.
Keeps the number of people handling the samples to a minimum to ensure proper field Chain-of-Custody.

Field Chain-of-Custody Records will accompany all analytical samples and sample shipping containers to document their transfer from the field to the analytical laboratory. The procedures to be implemented are as follows:

- Complete CRA supplied Chain-of-Custody Records indicating sample identification, containers filled, sampling date, sampling time, sample collector's name, and sample preservation, if applicable. Also note this information in the field notebooks.
- Repack shipping containers with samples, Chain-of-Custody Records, and water ice. Assign a Chain-of-Custody Record to each set of sample containers to be shipped.
- Place completed Chain-of-Custody Records in a plastic bag, seal the bag, and tape it to the inside cover of the shipping container. After the samples are iced, add the date to the Chain-of-Custody Record, seal the coolers with strapping tape, add custody seals, and ship the coolers to TA – NC, TA – KX or subcontractor laboratory using an overnight delivery service. Identify common carriers or intermediate individuals on the Chain-of-Custody Record, and retain copies of all bills-of-lading. When the samples are received in the laboratory, handle and process them in accordance with laboratory SOPs, or specified analytical methods, as defined in this QAPP.
- The laboratory receiving the samples will check shipping containers for completeness of paperwork, broken custody seals, damaged sample containers, and sample preservation as specified by the analytical method. The laboratory's sample management staff will note any problems, log the samples into the laboratory, and complete the Chain-of-Custody Record. The person relinquishing the samples to the facility or agency will request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is to be noted in the "Received By" space on the Record.
- Include copies of the Chain-of-Custody Record with the analytical data.

A separate sample receipt is prepared whenever samples are split with a government agency. The receipt is marked to indicate with whom the samples are being split. The person relinquishing the samples to the agency should request the agency representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is to be noted on the receipt and in the field notebook.

A copy of the Chain-of-Custody Record will accompany the samples to the laboratory. The field sampling personnel will retain one copy with the field notes. If a Chain-of-Custody Record is damaged in shipment, the field copy will be made available. A written statement will be prepared by the person who collected the samples, listing the samples that were recorded on the damaged record, and describing when and how the samples were collected. The statement should include information such as field notebook entries regarding the sample. This statement is submitted to Field QA Officer and the CRA Project Manager for further action, as necessary.

SAMPLE IDENTIFICATION PROCEDURE:

Label each bottle with the project number, the sample identifier, the sample type, the sampler's initials, and the date and time of sample collection. Complete sample labels for each sample and custody seals for each shipment container using waterproof ink, unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample tag because the ink pen would not function in freezing weather.

CHAIN-OF-CUSTODY PROCEDURES:

An example CRA Chain-of-Custody (COC) Record is shown in Appendix C. The CRA COC record will be used exclusively for all samples collected and submitted for environmental analysis. The COC Records should be legibly completed. Errors will be corrected by drawing a single line through the incorrect information and entering the correct information. All corrections are to be initialed and dated by the person making the correction. This procedure applies to words or figures inserted or added to a previously recorded statement.

CHAIN-OF-CUSTODY PROCEDURES *(continued)*:

The following information must be included on the Chain-of-Custody Record Appendix C:

- Facility name and address, project number, and sampler identification.
- "The Sample ID No. and Description" portion of the Record must be completed for each sample. This information includes the Field Sample ID, sample date and time, and sample depth. The sampling time MUST also be noted on the sample bottle (except for blind field duplicates, where date and time would not be noted on the bottle label or Chain-of-Custody Record).
- The sample container type and number, sample matrix, preservative/filtration, and requested analysis must be designated by checking the appropriate box and/or writing the required information.

Sample custody is documented on the lower portion of the Record, and includes the sampler's signature, signatures of persons involved in the possession of the sample with dates and times, and the date on which the sample was received at the laboratory, as described further below.

- Relinquished by/Received by - This part of the Chain-of-Custody Record is a record of the individuals who actually had the samples in their custody. The spaces must be used in chronological order as the Chain-of-Custody Record is transferred with the samples.
  1) Sampler signs when relinquishing custody.
  (1) Person accepting custody of samples from sampler signs.
  (2) Person in (1) must sign when relinquishing custody.
  (2)-(3) These are completed as necessary in the same manner as above.
- Sampler - The person/persons collecting the samples must sign their name and print their name under their signature, and record the date and time they relinquish the samples to either the laboratory or the shipper. The final signature is that of the person receiving the samples at the laboratory.
- Special Instructions – The sampler may provide additional information about a sample, e.g., if an odor is present, high or low pH, etc.
- Possible Hazard Identification – The sampler may include any known or suspected hazards associated with the samples. Sample entry personnel may add information to this section based on communications from the laboratory Project Manager or Supervisor after samples are received. Laboratory Team Leaders will use any hazard information to update and advise their analysts before work is started.

Note: If commercial carriers are used, the name of the carrier, any airbill number, and the date and time of relinquishing the sample containers are written on the airbill by sample entry or field personnel, and the airbill is attached to the Chain-of-Custody Record.

A copy of the Chain-of-Custody Record should be returned with the sample results. The laboratory service request number should be written on the Chain-of-Custody Record to facilitate its use during project data entry.

LABORATORY SAMPLING CUSTODY PROCEDURES (receipt of samples, archiving, disposal):

The laboratory assigns a unique, sequentially numbered sample code to each sample received. Laboratory custody procedures for sample receiving and log-in, storage, tracking, and holding time requirements are described in the laboratory's Quality Assurance Manual and in the Laboratory SOPs (see *Appendix D, Appendix E, and Appendix F).*

QAPP Worksheet #28 - Analytical Quality Control and Corrective Action

28-1 - MS-VOA / VOCs

Matrix: Solid, Water
Analytical Group: MS-VOA / VOCs
Analytical Method / SOP: NC-MS-019
Sampling SOP: FSP, Appendix J-F; QAPP, Appendix G
Analytical Organization: TA-NC
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.	Qualify data as needed	CRA QA Officer	RPD
Trip Blank	1 per cooler	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Check of mass spectral ion intensities (tuning procedure) using BFB (8260B)	Prior to initial calibration and calibration verification	Must meet the method requirements before samples are analyzed in accordance with DoD QSM requirements.	Retune instrument and verify the tune acceptability in accordance with DoD QSM requirements.	TestAmerica Analyst	Meets all EPA Method requirements
Internal standards	Every field sample, standard, and QC sample.	Areas within -50% to +100% of midpoint of the last ICAL for each sample and QC in accordance with DoD QSM requirements	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning in accordance with DoD QSM requirements.	TestAmerica Analyst	Meets all EPA Method requirements
Method blank	One per analytical batch (8260B)	No target analytes ≥ ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected >RL in accordance with DoD QSM requirements.	Verify instrument is clean, then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements.	TestAmerica Analyst	No target analytes ≥ 1/2 RL
Matrix Spike / Matrix Spike Duplicate	One MS/MSD per analytical /preparation batch	QSM or laboratory statistically derived control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	TestAmerica Analyst	QSM or laboratory statistically derived control limits

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Laboratory Control Sample	One LCS per analytical / preparation batch	QSM or laboratory statistically derived control limits in accordance with DoD QSM requirements.	Reanalyze LCS once. If acceptable, report. Otherwise, evaluate and re-prep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	TestAmerica Analyst	QSM or laboratory statistically derived control limits
Surrogate standards	All field and QC samples.	In accordance with DoD QSM criteria and requirements.	Evaluate matrix, then analytical data, then re-extract and reanalyze all affected samples in accordance with DoD QSM requirements as appropriate. Qualify outliers. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	TestAmerica Analyst	QSM or laboratory statistically derived control limits

28-2 - MSS-VOA / SVOCs

Matrix: Solid, Water
Analytical Group: MSS-VOA / SVOCs
Analytical Method / SOP: NC-MS-018, NC-OP-040, NC-OP-037
Sampling SOP: FSP, Appendix J-F; QAPP, Appendix G
Analytical Organization: TA-NC
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.	Qualify data as needed	CRA QA Officer	RPD
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit
Internal standards	During acquisition of calibration standard, samples, and QC check samples	Areas within -50% to +100% of midpoint of the last ICAL for each sample and QC in accordance with DoD QSM requirements.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning in accordance with DoD QSM requirements.	TestAmerica Analyst	Meets all EPA Method requirements

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Method blank	One per batch	No target analytes ≥ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected >RL in accordance with DoD QSM requirements.	Verify instrument is clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements.	TestAmerica Analyst	No target analytes ≥ RL
Matrix Spike / Matrix Spike Duplicate	One MS/MSD per batch	QSM or laboratory statistically derived control limits. RPD < 30% between MS and MSD.	Examine the project-specific DQOs. Contact the client as to the additional measures to be taken.	TestAmerica Analyst	QSM or laboratory statistically derived control limits
Laboratory Control Sample	One per preparatory batch	QSM or laboratory statistically derived control limits in accordance with DoD QSM requirements.	Reanalyze LCS once. If acceptable, report. Otherwise, evaluate and re-prepare and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	TestAmerica Analyst	QSM or laboratory statistically derived control limits
Surrogate standards	All field and QC samples.	In accordance with DoD QSM criteria and requirements.	Evaluate data, if preparation problem noted re-extract and reanalyze. Otherwise, qualify data in accordance with DoD QSM requirements.	TestAmerica Analyst	QSM or laboratory statistically derived control limits

28-3 - GC-ECD / Pesticides

Matrix: Water, Solid
Analytical Group: GC-ECD / Pesticides
Analytical Method / SOP: NC-GC-038, NC-OP-037, NC-OP-040
Sampling SOP: FSP, Appendix J-F; QAPP, Appendix G
Analytical Organization: TA-NC
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.	Qualify data as needed	CRA QA Officer	RPD
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Method Blank	One MB per analytical batch	No target analytes ≥ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected >RL in accordance with requirements.	Verify instrument is clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with requirements	Group Leader / Analyst	Bias
Laboratory Control Sample	One LCS per analytical batch	QC acceptance criteria as specified by laboratory statistically derived control limits in accordance with requirements	Reanalyze LCS once. If acceptable, report. Otherwise, evaluate and re-prepare and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Group Leader / Analyst	Accuracy
Surrogate Standards	Each field and QC sample	QC acceptance criteria as specified by laboratory statistically derived control limits in accordance with requirements	For QC and field samples, correct problem, then re-prepare and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Group Leader / Analyst	Bias, accuracy, and precision

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Matrix Spike / Matrix Spike Duplicates	One MS / MSD pair per analytical batch	QC acceptance criteria as specified by laboratory statistically derived control limits in accordance with requirements. RPD < 30% between MS and MSD.	Examine the project specific DQOs. Evaluate the data. Contact the client as to additional measures to be taken.	Group Leader / Analyst	Accuracy and precision

28-4 - GC-ECD / Herbicides

Matrix: Water, Solid
Analytical Group: GC-ECD / Herbicides
Analytical Method / SOP: NC-GC-038, NC-OP-031
Sampling SOP: FSP, Appendix J-F; QAPP, Appendix G
Analytical Organization: TA-NC
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.	Qualify data as needed	CRA QA Officer	RPD
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Method Blank	One per preparation batch	No target analytes ≥ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected >RL.	Verify instrument is clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank.	Group Leader / Analyst	Accuracy/Bias Contamination
Laboratory Control Sample	One LCS per preparation batch	QC acceptance criteria as specified by laboratory statistically derived control limits.	Reanalyze LCS once. If acceptable, report. Otherwise, evaluate re-prepare and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Group Leader / Analyst	Precisions and Accuracy/Bias

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Surrogates	Each field and QC sample	QC acceptance criteria as specified by laboratory statistically derived control limits.	For QC and field samples, correct problem, then re-prepare and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Group Leader / Analyst	Precisions and Accuracy/Bias
Matrix Spike / Matrix Spike Duplicate	One MS / MSD pair per preparation batch	QC acceptance criteria as specified by laboratory statistically derived control limits. RPD < 30% between MS and MSD.	Examine the project specific DQOs. Evaluate the data. Contact the client as to additional measures to be taken.	Group Leader / Analyst	Precisions and Accuracy/Bias

28-5 - GCS, GCV / Dissolved Gas

Matrix: Water
Analytical Group: GCS, GCV / Dissolved Gas
Analytical Method / SOP: NC-GC-032
Sampling SOP: FSP, Appendix J-F; QAPP, Appendix G
Analytical Organization: TA-NC
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL.	Qualify data as needed	CRA QA Officer	RPD
Trip Blank	1 per cooler	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit
Method Blank	One per analytical batch	No results ≥ RL	Verify instrument is clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with requirements.	Group Leader / Analyst	Bias

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Laboratory Control Sample	One per analytical batch	QC acceptance criteria as specified by laboratory statistically derived control limits in accordance with requirements	Terminate analysis, identify and correct the problem, then re-prepare and reanalyze all affected samples and QC checks in accordance with requirements.	Group Leader / Analyst	Accuracy
Matrix Spike / Matrix Spike Duplicate	One pair per analytical batch (water matrix only)	QC acceptance criteria as specified by laboratory statistically derived control limits in accordance with requirements	Examine the project specific DQOs. Evaluate the data, and re-prepare/reanalyze the native sample and MS/MSD pair as indicated.	Group Leader / Analyst	Precision
Sample Duplicate	One per every 10 client samples (solid matrix only)	RPD $\leq$ 20%	Examine sample homogeneity, re-prepare and reanalyze sample and duplicate.	Group Leader / Analyst	Precision

28-6 - ICP/ Metals

Matrix: Solid, Water
Analytical Group: ICP/ Metals
Analytical Method / SOP: NC-IP-010, NC-IP-011, NC-MT-012
Sampling SOP: FSP, Appendix J-F; QAPP, Appendix G
Analytical Organization: TA-NC
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.	Qualify data as needed	CRA QA Officer	RPD
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Method Blank	One per digestion batch	No target analytes ≥ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected >RL.	Verify instrument is clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank.	Group Leader / Analyst	Bias
Laboratory Control Sample	One LCS per each preparation batch	QC acceptance criteria: laboratory statistically derived control limits.	Evaluate LCS data and reanalyze if bias appears instrument related. If bias appears preparation related, determine if trend requires correction prior to re-prepare and reanalysis of the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Group Leader / Analyst	Accuracy
Matrix Spike / Matrix Spike Duplicate	One MS/MSD pair per preparation batch	QC acceptance criteria: laboratory statistically derived control limits and RPD ≤ 20%.	Examine the project specific DQOs. Evaluate the data, and re-prepare/reanalyze the native sample and MS/MSD pair as indicated.	Group Leader / Analyst	Precision
Dilution Test	Each new sample matrix	1:5 dilution must agree within ±10% of the original determination.	Perform post-digestion spike addition.	Group Leader / Analyst	Precision

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Post digestion Spike Addition	When dilution test fails or analyte concentration in all samples < 50 x RL.	Recovery within 75% to 125% of expected results.	Flag for matrix interference.	Group Leader / Analyst	Precision

28-7 - CVAA / Mercury

Matrix: Water, Solid
Analytical Group: CVAA / Mercury
Analytical Method / SOP: NC-MT-014
Sampling SOP: FSP, Appendix J-F; QAPP, Appendix G
Analytical Organization: TA-NC
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.	Qualify data as needed	CRA QA Officer	RPD
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Method Blank	One per prep batch	No target analytes $\geq$ RL in accordance with requirements	Verify instrument is clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with requirements.	Group Leader / Analyst	Bias
Laboratory Control Sample	One per prep batch	QC acceptance criteria: 81% to 123% (waters) or 73% to 121% (solids) accuracy, 20% precision or laboratory statistically derived control limits	Terminate analysis, identify and correct the problem, then re-prepare and reanalyze all affected samples and QC checks in accordance with requirements.	Group Leader / Analyst	Accuracy
Matrix Spike / Matrix Spike Duplicate	One MS / MSD pair per prep batch	QC acceptance criteria: 69% to 134% (waters) and 11% to 192% (solids) accuracy, 20% precision or laboratory statistically derived control limits	Examine the project specific DQOs. If the matrix spike falls outside of criteria, additional quality control tests are required to evaluate matrix effects.	Group Leader / Analyst	Precision

28-8 - Cyanide

Matrix: Water, Solid
Analytical Group: Cyanide
Analytical Method / SOP: NC-WC-031, NC-WC-032
Sampling SOP: FSP, Appendix J-F; QAPP, Appendix G
Analytical Organization: TA-NC
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.	Qualify data as needed	CRA QA Officer	RPD
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit
Method Blank	One per analytical batch	No target analyte ≥ RL	Evaluate blank to determine if instrument or solution caused, then correct. Re-prepare and reanalyze the blank. All samples following the last acceptable calibration blank must be reanalyzed.	Group Leader / Analyst	Bias

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Laboratory Control Sample	One per analytical batch	QC acceptance criteria as specified by laboratory statistically derived control limits in accordance with requirements	Terminate analysis, identify and correct the problem, then re-prepare and reanalyze all affected samples and QC checks in accordance with requirements.	Group Leader / Analyst	Accuracy and Bias
Matrix Spike / Matrix Spike Duplicate	One MS/MSD pair per analytical batch	QC acceptance criteria as specified by laboratory statistically derived control limits in accordance with requirements	Examine the project specific DQOs. Evaluate the data, and re-prepare/reanalyze the native sample and MS/MSD pair as indicated.	Group Leader / Analyst	Precision and Accuracy

28-9 - General Chemistry / Total Solids

Matrix: Solid and Water
Analytical Group: General Chemistry / Total Solids
Analytical Method / SOP: NC-WC-004
Sampling SOP: FSP, Appendix J-F; QAPP, Appendix G
Analytical Organization: TA-NC
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.	Qualify data as needed	CRA QA Officer	RPD
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit
Sample Duplicate	One per every 10 samples	RPD ≤ 20%	Re-analyze sample	Group Leader / Analyst	Precision

28-10 - Wet Chemistry / Alkalinity

Matrix: Water
Analytical Group: Wet Chemistry / Alkalinity
Analytical Method / SOP: NC-WC-093
Sampling SOP: FSP, Appendix J-F; QAPP, Appendix G
Analytical Organization: TA-NC
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.	Qualify data as needed	CRA QA Officer	RPD
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit
Method Blank	One per analytical batch	No target analyte > RL	Riprap and reanalyze batch. Any samples < RL may be reported and narrated.	Group Leader / Analyst	Bias
Laboratory Control Sample	One per analytical batch	QC acceptance criteria as specified by laboratory statistically derived control limits in accordance with requirements	Riprap and reanalyze batch. If LCS is outside of acceptance on the high side, any sample that is < RL may be reported if all other QC passes criteria.	Group Leader / Analyst	Accuracy and Bias
Matrix Spike / Matrix Spike	One MS / MSD pair per analytical batch	QC acceptance criteria as specified by laboratory	If RPD ≤ 20%, and LCS meets criteria, samples may be	Group Leader / Analyst	Accuracy and Precision

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Duplicate (total alkalinity)	for Total Alkalinity	statistically derived control limits in accordance with requirements	reported and narrated. Otherwise, associated samples must be re-prepped and reanalyzed.		
Sample Duplicate	One for every 10 samples	RPD $\geq$ 20%	Re-prep and reanalyze sample and associated samples.	Group Leader / Analyst	Accuracy and Precision

28-11 - Wet Chemistry / Anions

Matrix: Water, Solid
Analytical Group: Wet Chemistry / Anions
Analytical Method / SOP: NC-WC-084
Sampling SOP: FSP, Appendix J-F; QAPP, Appendix G
Analytical Organization: TA-NC
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.	Qualify data as needed	CRA QA Officer	RPD
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Method Blank	One per analytical batch	No analytes detected >RL in accordance with requirements.	Verify instrument is clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with requirements.	Group Leader / Analyst	Bias
Laboratory Control Sample	One per analytical batch	QC acceptance criteria as specified by laboratory statistically derived control limits in accordance with requirements	Terminate analysis, identify and correct the problem, then re-prepare and reanalyze all affected samples and QC checks in accordance with requirements.	Group Leader / Analyst	Accuracy and Bias
Matrix Spike / Matrix Spike Duplicate	One MS / MSD pair per analytical batch	QC acceptance criteria as specified by laboratory statistically derived control limits in accordance with requirements. RPD less than or equal to 15%.	Examine the project specific DQOs. Evaluate the data, and re-prepare/reanalyze the native sample and MS / MSD pair as indicated.	Group Leader / Analyst	Precision and Accuracy

28-12 - Wet Chemistry / TOC

Matrix: Water and Solid
Analytical Group: Wet Chemistry / TOC
Analytical Method / SOP: NC-WC-017, NC-WC--18
Sampling SOP: FSP, Appendix J-F; QAPP, Appendix G
Analytical Organization: TA-NC
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.	Qualify data as needed	CRA QA Officer	RPD
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Method Blank	One per analytical batch	No results $\geq$ RL	Verify instrument is clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with requirements.	Group Leader / Analyst	Bias
Laboratory Control Sample	One per analytical batch	QC acceptance criteria as specified by laboratory statistically derived control limits in accordance with requirements	Terminate analysis, identify and correct the problem, then re-prepare and reanalyze all affected samples and QC checks in accordance with requirements.	Group Leader / Analyst	Accuracy
Matrix Spike / Matrix Spike Duplicate	One pair per analytical batch (water matrix only)	QC acceptance criteria as specified by laboratory statistically derived control limits in accordance with requirements	Examine the project specific DQOs. Evaluate the data, and re-prepare/reanalyze the native sample and MS/MSD pair as indicated.	Group Leader / Analyst	Precision
Sample Duplicate	One per every 10 client samples (solid matrix only)	RPD $\leq$ 20%	Examine sample homogeneity, re-prepare and reanalyze sample and duplicate.	Group Leader / Analyst	Precision

28-13 - Wet Chemistry / pH (Corrosivity)

Matrix: Water, Soil
Analytical Group: Wet Chemistry / pH (Corrosivity)
Analytical Method / SOP: NC-WC-010
Sampling SOP: FSP, Appendix J-F; QAPP, Appendix G
Analytical Organization: TA-NC
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.	Qualify data as needed	CRA QA Officer	RPD
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit
Laboratory Control Sample	One per analytical batch	Recovery criteria of 97-103%	Trouble shoot probe, refresh buffers, recalibrate	Group Leader / Analyst	Accuracy
Sample Duplicate	One per every 10 client samples	RPD ≤ 20%	Re-aliquot and reanalyze	Group Leader / Analyst	Precision and accuracy

28-14 - Wet Chemistry / Sulfide

Matrix: Water, Solid
Analytical Group: Wet Chemistry / Sulfide
Analytical Method / SOP: NC-WC-060
Sampling SOP: FSP, Appendix J-F; QAPP, Appendix G
Analytical Organization: TA-NC
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.	Qualify data as needed	CRA QA Officer	RPD
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit
Method Blank	One per analytical batch (and matrix for 9034).	No result ≥ RL	Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with requirements.	Group Leader / Analyst	Bias

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Laboratory Control Sample	One per analytical batch (and matrix for 9034).	QC acceptance criteria as specified by laboratory statistically derived control limits in accordance with requirements	Terminate analysis, identify and correct the problem, then re-prepare and reanalyze all affected samples and QC checks in accordance with requirements.	Group Leader / Analyst	Accuracy
Matrix Spike / Matrix Spike Duplicate	One per analytical batch (and matrix for 9034).	QC acceptance criteria as specified by laboratory statistically derived control limits in accordance with requirements	Examine the project specific DQOs. Evaluate the data, and re-prepare/reanalyze the native sample and MS/MSD pair as indicated.	Group Leader / Analyst	Precision

28-15 - General Chemistry / Hardness

Matrix: Water
Analytical Group: General Chemistry / Hardness
Analytical Method / SOP: NC-WC-036
Sampling SOP: FSP, Appendix J-F; QAPP, Appendix G
Analytical Organization: TA-NC
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.	Qualify data as needed	CRA QA Officer	RPD
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit
Method Blank	One per analytical batch and matrix	No analytes > RL	Verify glassware cleanliness and reagent prep dates. Re-prepare and reanalyze batch.	Group Leader / Analyst	Bias
Laboratory Control Sample	One per analytical batch and matrix	QC acceptance criteria as specified by laboratory statistically derived control limits in accordance with requirements	Terminate analysis, identify and correct the problem, then re-prepare and reanalyze all affected samples and QC checks in accordance with requirements	Group Leader / Analyst	Accuracy

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Matrix Spike / Matrix Spike Duplicate	One per analytical batch and matrix	QC acceptance criteria as specified by laboratory statistically derived control limits in accordance with requirements	Examine the project specific DQOs. Evaluate the data, and re-prepare/reanalyze the native sample and MS/MSD pair as indicated.	Group Leader / Analyst	Precision and Accuracy
Sample Duplicate	One for every 10 samples	RPD ≤ 20%	Examine the project specific DQOs. Evaluate the data, and re-prepare/reanalyze the native sample and duplicate as indicated.	Group Leader / Analyst	Precision

28-16 - Wet Chemistry / Flashpoint

Matrix: Solid and Water
Analytical Group: Wet Chemistry / Flashpoint
Analytical Method / SOP: NC-WC-034
Sampling SOP: FSP, Appendix J-F; QAPP, Appendix G
Analytical Organization: TA-NC
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.	Qualify data as needed	CRA QA Officer	RPD
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit
Laboratory Control Sample	One p-Xylene check per analytical batch	81 °F ± 2 °F	Troubleshoot instrument, cool p-Xylene, reanalyze	TestAmerica Analyst	Accuracy
Sample Duplicate	One per matrix	≥ 20% RPD	Analyze a third aliquot	TestAmerica Analyst	Precision and Accuracy

28-17 - Dioxins and Furans

Matrix: Water, Solid
Analytical Group: Dioxins and Furans
Analytical Method / SOP: WS-ID-0005, WS-IDP-0005
Sampling SOP: FSP, Appendix J-F; QAPP, Appendix G
Analytical Organization: TA-NC
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Water: RPD ≤ 50% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 1X RL. Soil: RPD ≤ 100% for sample results that are > 5X RL; or for sample results that are < 5X RL, the absolute difference of the two results is less than 2X RL.	Qualify data as needed	CRA QA Officer	RPD
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit
Method Blank	One per preparation batch	Project specific criteria, if available. Otherwise, no target analytes detected ≥ 1/2 RL or ≥ 20% of the associated regulatory limit or ≥ 5% of the sample result for the analyte, whichever is greater. (OCDD is considered a common laboratory contaminant and treated accordingly).	Verify instrument is clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the	Chemist	No target analytes ≥ Reporting Limit.

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
			contaminated blank in accordance with DoD QSM requirements. "Totals" are not considered "target analytes" – no corrective action or flagging is necessary for "totals".		
Internal Standard Spike	Every field sample, standard and QC sample	% recovery for each IS in the original sample (prior to dilutions) must be within 40 - 135% recovery.	Correct problem, then re-prepare and reanalyze the samples with failed IS.	Lab Manager / Analyst	Meets all EPA Method requirements (40-135% Recovery)
Laboratory Control Sample (LCS)	One per sample preparation batch	Laboratory statistically derived control limits	Reanalyze LCS once. If acceptable, report. Otherwise, evaluate for impact (high bias and non-detects, or sporadic marginal exceedence may be narrated and reported). If impact too great, re-prepare and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Lab Manager / Analyst	Laboratory statistically derived control limits
Matrix Spike / Matrix Spike Duplicate	One MS / MSD per analytical / preparation batch (if requested by client).	Laboratory statistically derived control limits, RPD ≤ 20%.	Identify problem; if not related to matrix interference, re-extract and reanalyze field sample and MS/MSD, provided sufficient sample material is available.	Lab Manager / Analyst	Laboratory statistically derived control limits

28-18 - Volatile Organics

Matrix: Air
Analytical Group: Volatile Organics
Analytical Method / SOP: KNOX-MS-0001
Sampling SOP: FSP, Appendix J-F
Analytical Organization: TA-KX
Sampler's Name: TBD
Field Sampling Organization: TBD
No. of Sample Locations: TBD

QC SAMPLE	*FREQUENCY/NUMBER*	*METHOD/SOP ACCEPTANCE CRITERIA*	*CORRECTIVE ACTION*	*TITLE/POSITION OF PERSON RESPONSIBLE FOR CORRECTIVE ACTION*	*PROJECT-SPECIFIC MPC*
Field duplicate	1 per 10 samples	Advisory limit of RPD ≤ 50% for air samples	Qualify data as needed	CRA QA Officer	RPD
Field / Equipment Blank	1 per day	< Laboratory reporting limit	Qualify data as needed	CRA QA Officer	< Laboratory reporting limit
Sample Duplicate	One per every 10 samples	RPD ≤ 20%	Re-analyze sample	Group Leader / Analyst	Precision
Method Blank	1 per 20 samples or 24 hr tune, whichever is more frequent	No Target Compounds>=RL	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >20x blank result or sample results ND.	Analyst / Section Supervisor	No Target Compounds>=RL
Laboratory Control Sample	1 per 20 samples or 24 hr tune, whichever is more frequent	70-130% recovery with provisory analytes within 60-140%. Marginal exceedence limit of 60-140 % / 50-150 % allowed based on # of target analytes	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst / Section Supervisor	Laboratory % Recovery Control Limits.
Laboratory Duplicate	1/Batch (20 samples)	RPD ≤25 for analytes >5x RL	Determine root cause; reanalyze DUP; flag data; discuss in narrative.	Analyst / Section Supervisor	Laboratory % Recovery / RPD Control Limits

QAPP Worksheet #29 - Project Documents and Records

SAMPLE COLLECTION DOCUMENTS AND RECORDS	*ON-SITE ANALYSIS DOCUMENTS AND RECORDS*	*OFF-SITE ANALYSIS DOCUMENTS AND RECORDS*	*DATA ASSESSMENT DOCUMENTS AND RECORDS*	*OTHER*
• Field notes • Sampling logs • Chain-of-Custody Records • Air bills • Custody Seals • Communication logs	• Equipment calibration logs • Field data records • Soil stratigraphy logs	• Sample receipt, custody, and tracking records • Standard traceability logs • Sample prep logs • Run logs • Equipment maintenance, testing, and inspection logs • Corrective action forms • Reported field sample results • Reported results for standards, QC checks, and QC samples • Instrument printouts (raw data) for field sample standards, QC checks, and QC sample • Data package completeness checklists • Sample disposal records • Extraction/Cleanup records • Raw data (stored on diskette or CD-R) • Analytical reports	• Data validation checklists • Data quality assessments	• Consent Decree documents • Progress reports to the U.S. EPA • Work plans and Field Sampling Plans • Health and Safety Plans • Quality Assurance Project Plan • Quality Management Plan • Remedial Investigation and Risk Assessment Reports • Feasibility Studies • Design Reports • Monitoring Reports

QAPP Worksheet #30 - Analytical Services

MATRIX	*ANALYTICAL GROUP*	*CONCENTRATION LEVEL*	*SAMPLE LOCATIONS/ID NUMBERS*	*ANALYTICAL SOP*	*DATA PACKAGE TURNAROUND TIME*	*LABORATORY/ORGANIZATION (name, address, contact, telephone number)*	*BACKUP LABORATORY/ORGANIZATION (name, address, contact, telephone number)*
Groundwater	VOCs	All	TBD	D-1		TestAmerica, Laboratories, Inc. 4101 Shuffel Street NW North Canton, OH 44720 Denise Heckler 330-996-9477	
	SVOCS			D-2, E-1			
	PCBs			D-3, E-1			
	Metals			D-5, E-9			
	Cyanide			D-7, E-5			
	Mercury			D-6			
	Pesticides			D-3, E-1			
	Herbicides			D-3, E-7			
	Dioxin&Furan			D-16, E-6			
	Alkalinity			D-9			
	Anions			D-10			
	TOC			D-11			
	pH			D-12			
	Sulfide			D-13			
	Dissolved Gas			D-4			
	Hardness			D-14			

MATRIX	*ANALYTICAL GROUP*	*CONCENTRATION LEVEL*	*SAMPLE LOCATIONS/ID NUMBERS*	*ANALYTICAL SOP*	*DATA PACKAGE TURNAROUND TIME*	*LABORATORY/ORGANIZATION (name, address, contact, telephone number)*	*BACKUP LABORATORY/ORGANIZATION (name, address, contact, telephone number)*
Soil	VOCs SVOCS PCBs Metals Cyanide Mercury Pesticides Herbicides Dioxin&Furan pH Sulfide Total Solids TOC	All	TBD	D-1 D-2, E-4 D-3, E-4 D-5, E-8 D-7, E-5 D-6 D-3, E-4 D-3, E-7 D-16, E-6 D-12 D-13 D-8 D-18		TestAmerica, Laboratories, Inc. 4101 Shuffel Street NW North Canton, OH 44720 Denise Heckler 330-996-9477	
Waste Characterization (TCLP Solids and Water)	TCLP: VOCs, SVOCs, Metals, Cyanide, Hg, Pesticides, Herbicides, pH, Sulfide, Flashpoint	All	TBD	E-10		TestAmerica, Laboratories, Inc. 4101 Shuffel Street NW North Canton, OH 44720 Denise Heckler 330-996-9477	

MATRIX	*ANALYTICAL GROUP*	*CONCENTRATION LEVEL*	*SAMPLE LOCATIONS/ID NUMBERS*	*ANALYTICAL SOP*	*DATA PACKAGE TURNAROUND TIME*	*LABORATORY/ORGANIZATION (name, address, contact, telephone number)*	*BACKUP LABORATORY/ORGANIZATION (name, address, contact, telephone number)*
Air / Soil Gas	VOCs	Low / Standard		D-17		TestAmerica, Laboratories, Inc 5815 Middlebrook Pike Knoxville, TN 37921 Jamie McKinney 865-291-3051	

QAPP Worksheet #31 - Planned Project Assessments

ASSESSMENT TYPE	*FREQUENCY*	*INTERNAL OR EXTERNAL*	*ORGANIZATION PERFORMING ASSESSMENT*	*PERSON(S) RESPONSIBLE FOR PERFORMING ASSESSMENT*	*PERSON(S) RESPNSIBLE FOR RESPONDING TO ASSESSMENT FINDINGS*	*PERSON(S) RESPONSIBLE FOR IDENTIFYING AND IMPLEMENTING CORRECTIVE ACTION*	*PERSON(S) RESPONSIBLE FOR MONITORING EFFECTIVENESS OF CORRECTIVE ACTION*
Field performance audit	Periodic, based on field schedule	Internal	CRA	Steve Quigley CRA (Project Manager)	Steve Quigley, (or as assigned) CRA (Project Manager)	Greg Lewis, (or as assigned) CRA (Field QA Officer)	Steve Quigley, (or as assigned) CRA (Project Manager)
Field systems audit	Periodic, based on field schedule	Internal	CRA	Angela Bown CRA (QA Officer)	Steve Quigley, (or as assigned) CRA (Project Manager)	Greg Lewis, (or as assigned) CRA (Field QA Officer)	Steve Quigley, (or as assigned) CRA (Project Manager)
Laboratory audit	As needed, based on laboratory performance	Internal	CRA	Angela Bown CRA (QA Officer)	Denise Heckler TA-NC (Laboratory Project Manager)	Denise Heckler TA-NC (Laboratory Project Manager)	Angela Bown CRA (QA Officer)
Laboratory audit	Per laboratory QA Plan	Internal	TA-NC	Dorothy Leeson TA-NC (Laboratory QA Officer)	Denise Heckler TA-NC (Laboratory Project Manager)	Dorothy Leeson TA-NC (Laboratory QA Officer)	Angela Bown CRA (QA Officer)
Laboratory audit	Per laboratory QA Plan	Internal	TA – KX	Kevin McGee TA-KX (Laboratory QA Officer)	Jamie McKinney TA-KX (Laboratory Project Manager)	Kevin McGee TA-KX (Laboratory QA Officer)	Angela Bown CRA (QA Officer)
QAPP	Annually	Internal	CRA	Steve Quigley CRA (Project Manager)	Steve Quigley CRA (Project Manager)	Steve Quigley CRA (Project Manager)	USEPA

QAPP Worksheet #32 - Assessment Findings and Corrective Action Responses

ASSESSMENT TYPE	*NATURE OF DEFICIENCIES DOCUMENTATION*	*INDIVIDUAL(S) NOTIFIED OF FINDINGS*	*TIME FRAME OF NOTIFICATION*	*NATURE OF CORRECTIVE ACTION RESPONSE DOCUMENTATION*	*INDIVIDUAL(S) RECEIVING CORRECTIVE ACTION RESPONSE*	*TIME FRAME FOR RESPONSE*
Field performance audit	Checklist	Greg Lewis, CRA (Field QA Officer) will notify Steve Quigley, CRA (Project Manager)	Within 72 hours after audit (or sooner, as appropriate)	E-mail response	Greg Lewis (or as assigned), CRA (Field QA Officer)	Within 48 hours after notification (or sooner, as appropriate)
Field systems audit	Checklist	Angela Bown, CRA (QA Officer) will notify Steve Quigley, CRA (Project Manager)	Within 48 hours after audit (or sooner, as appropriate)	E-mail response	Greg Lewis (or as assigned), CRA (Field QA Officer)	Within 48 hours after notification (or sooner, as appropriate)
Internal laboratory audit TA-NC	Executive Summary from Management Report	Denise Heckler, TA-NC (Laboratory Project Manager) will notify Angela Bown, CRA (QA Officer), and appropriate laboratory staff	Within 48 hours after audit (or sooner, as appropriate)	Executive Summary from Management Report	Angela Bown, CRA (QA Officer), and appropriate laboratory staff	Within 48 hours after Notification (or sooner, as appropriate)
External laboratory audit TA-NC	Checklist	Angela Bown, CRA (QA Officer) will notify Denise Heckler, TA-NC (Laboratory Project Manager) and Steve Quigley, CRA (Project Manager)	Within 1 week after audit	Memorandum	Denise Heckler, TA-NC (Laboratory Project Manager)	Within 48 hours after notification (or sooner, as appropriate)
Internal laboratory audit TA – KX	Executive Summary from Management Report	Jamie McKinney, TA-KX (Laboratory Project Manager) will notify Angela Bown, CRA (QA Officer), and appropriate laboratory staff	Within 48 hours after audit (or sooner, as appropriate)	Executive Summary from Management Report	Angela Bown, CRA (QA Officer), and appropriate laboratory staff	Within 48 hours after notification (or sooner, as appropriate)

ASSESSMENT TYPE	*NATURE OF DEFICIENCIES DOCUMENTATION*	*INDIVIDUAL(S) NOTIFIED OF FINDINGS*	*TIME FRAME OF NOTIFICATION*	*NATURE OF CORRECTIVE ACTION RESPONSE DOCUMENTATION*	*INDIVIDUAL(S) RECEIVING CORRECTIVE ACTION RESPONSE*	*TIME FRAME FOR RESPONSE*
External laboratory audit TA – KX	Checklist	Angela Bown, CRA (QA Officer) will notify Jamie McKinney, TA-KX (Laboratory Project Manager) and Steve Quigley, CRA (Project Manager)	Within 1 week after audit	Memorandum	Jamie McKinney, TA-KX (Laboratory Project Manager)	Within 48 hours after notification (or sooner, as appropriate)

QAPP Worksheet #33 - QA Management Reports

TYPE OF REPORT	*FREQUENCY (daily, weekly, monthly, quarterly, annually, etc.)*	*PROJECTED DELIVERY DATE(S)*	*PERSON(S) RESPONSIBLE FOR REPORT PREPARATION (title and organizational affiliation)*	*REPORT RECIPIENT(S) (title and organizational affiliation)*
Field audit reports	As needed	As generated	Angela Bown, CRA (QA Officer)	Steve Quigley, CRA (Project Manager)
TA-NC audit (external)	As needed	As generated	Angela Bown, CRA (QA Officer)	Denise Heckler, TA-NC (Laboratory Project Manager) Steve Quigley, CRA (Project Manager)
TA-KX audit (external)	As needed	As generated	Angela Bown, CRA (QA Officer)	Jamie McKinney, TA-KX (Laboratory Project Manager) Steve Quigley, CRA (Project Manager)
Data validation reports	As specified in data assessment section	As generated	Angela Bown, CRA (QA Officer)	Steve Quigley, CRA (Project Manager)
Data quality summary	As appropriate for data use	As generated	Angela Bown, CRA (QA Officer)	Steve Quigley, CRA (Project Manager)
Final Project Report	As specified in Project Schedule	As generated	Steve Quigley, CRA (Project Manager)	Leslie Patterson, USEPA Region 5 (Remedial Project Manager) Steve Quigley, CRA (Project Manager)

QAPP Worksheet #34: Data Verification and Validation Inputs

ITEM	*DESCRIPTION*	*VERIFICATION (completeness)*	*VALIDATION (conformance to specifications)*
Planning Documents / Records			
1	Approved QAPP	X	
2	Contract	X	
3	Field SOPs	X	
4	Laboratory SOPs	X	
Field Records			
5	Field logbooks	X	X
6	Equipment calibrations records	X	X
7	Chain-of-Custody Forms	X	X
8	Sampling diagrams/surveys	X	X
9	Drilling Logs	X	X
10	Geophysics reports	X	X
11	Relevant Correspondence	X	X
12	Change orders/deviations	X	X
13	Field audit reports	X	X
14	Field corrective action	X	X

ITEM	DESCRIPTION	VERIFICATION (completeness)	VALIDATION (conformance to specifications)
	Analytical Data Package		
15	Cover sheet (laboratory identifying information)	X	X
16	Case narrative	X	X
17	Internal laboratory Chain-of-Custody	X	X
18	Sample receipt records	X	X
19	Sample chronology (i.e. dates and times of receipt, preparation, & analysis)	X	X
20	Communication records	X	X
21	Project-specific PT sample results	X	X
22	LOD/LOQ establishment and verification	X	X
23	Standards Traceability	X	X
24	Instrument calibration records	X	X
25	Definition of laboratory qualifiers	X	X
26	Results reporting forms	X	X
27	QC sample results	X	X
28	Corrective action reports	X	X
29	Raw data	X	X
30	Electronic data deliverable	X	X

QAPP Worksheet #35 - Data Verification and Validation Procedures

35-1 - Verification Process

VALIDATION INPUT	*DESCRIPTION*	*INTERNAL/EXTERNAL*	*RESPONSIBLE FOR VERIFICATION (name, organization)*
Chain-of-Custody Records and shipping documentation	Chain-of-Custody Records and shipping documentation will be reviewed by the laboratory upon receipt of samples for verification against the sample coolers they represent. The Chain-of-Custody Record will be signed by all parties who had custody of samples, with the exception of commercial carriers.	Internal	Denise Heckler, TA-NC (or designee) (Laboratory Project Manager) and/or Jamie McKinney, TA-KX) (Laboratory Project Manager)
Field notes and sampling logs	All field notes and sampling logs will be reviewed internally and placed in the project file.	Internal	Steve Quigley, CRA (Project Manager)
Laboratory analytical data package	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal.	Internal	Denise Heckler, TA-NC (or designee) (Laboratory Project Manager) and/or Jamie McKinney, TA-KX (Laboratory Project Manager)
Final Sample Report	All final data packages will be verified for content and sample information upon receipt. Data validation reports and outputs of the database will be used to prepare project submissions.	Internal	Angela Bown, CRA (QA Officer)
Electronic Data Deliverables (EDDs)	Determine whether required fields and format were provided compatible with EQUiS	Internal	Tim Harris, CRA (Data Manager)

35-2 - Validation Process

VALIDATION INPUT	*DESCRIPTION*	*RESPOSIBLE FOR VALIDATION (Name, Organization)*
Data Deliverables	Ensure that all required documentation on sampling and analysis was provided.	Angela Bown (or designee), CRA
Analytes	Ensure that the required list of analytes was reported as specified in governing documents	Angela Bown (or designee), CRA
Chain-of-Custody	Examine the traceability of the data from the time of sample collection until reporting of data. Examine COC records against contract, method, and QAPP.	Angela Bown (or designee), CRA
Holding Times	Identify holding time criteria and determine if they were met. If holding times were not met, confirm that deviations were documented.	Angela Bown (or designee), CRA
Sample Handling	Ensure that required sample handling, receipt, and storage procedures were followed and that any deviations were documented.	Angela Bown (or designee), CRA
Sampling Methods and Procedures	Ensure that all sampling SOPs were followed and that any deviations were noted.	Angela Bown (or designee), CRA
Field Transcription	Authenticate transcription accuracy for sampling data (i.e. from field notebook to reports.)	Angela Bown (or designee), CRA
Analytical Methods and Procedures	Establish that required analytical methods were used and that any deviations were noted. Determine if the QC samples met performance criteria and ensure that any deviations were noted.	Angela Bown (or designee), CRA
Data Qualifiers	Determine that the laboratory qualifiers were defined and applied as specified in methods, procedures, or contracts	Angela Bown (or designee), CRA
Laboratory Transcription	Authenticate accuracy of the transcription of analytical data (i.e., lab notebook to report form, or instrument to LIMS).	Angela Bown (or designee), CRA
Verification of Calculations	Verify 5% of all calculations summarized on the laboratory's QA/QC summary sheets.	Angela Bown (or designee), CRA
Standards	Determine if standards are traceable and meet contract, method, or procedural requirements.	Angela Bown (or designee), CRA
Deviations	Determine the impacts of any deviations from sampling or analytical methods and SOPs.	Angela Bown (or designee), CRA
Documentation of Method QC Results	Determine if all method required QC samples were analyzed and met required acceptance limits.	Angela Bown (or designee), CRA

VALIDATION INPUT	*DESCRIPTION*	*RESPOSIBLE FOR VALIDATION (Name, Organization)*
Project Quantitation Limits	Determine if all sample results met the project quantitation limits specified in the QAPP.	Angela Bown (or designee), CRA
Field Duplicates	Compare results of field duplicates with criteria established in the QAPP.	Angela Bown (or designee), CRA
Performance Criteria	Evaluate QC data against project-specific performance criteria in the QAPP.	Angela Bown (or designee), CRA
Data Qualifiers	Determine that the data qualifiers are appropriate and justified.	Angela Bown (or designee), CRA
Data Validation Report	Summarize deviations from the methods, procedures, or contracts. Summarize the outcome of comparison of data to performance criteria in the QAPP. Include qualified data and explanation of all data qualifiers.	Angela Bown (or designee), CRA

QAPP Worksheet #36 - Validation Summary

MATRIX	*ANALYTICAL GROUP*	*CONCENTRATION LEVEL*	*VALIDATION CRITERIA*	*DATA VALIDATOR (title and organizational affiliation)*
Water/Solid	VOCs, SVOCs PCBs, Pesticides, Herbicides,	Low, medium, high	"USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review", United States Environmental Protection Agency (USEPA) 540-R-8-01, June 2008; Method and laboratory SOP criteria, QAPP criteria and professional judgment	Angela Bown (or designee), CRA
Water/Solid	Metals, Mercury, Cyanide, Total Alkalinity, Anions, DOC, Dissolved Gases, Hardness, Sulfide, pH	Low, medium, high	"USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review", USEPA 540-R-10-011, January 2010; Method and laboratory SOP criteria, QAPP criteria and professional judgment	Angela Bown (or designee), CRA
Air/Soil Gas	VOCs	Standard	"USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review", United States Environmental Protection Agency (USEPA) 540-R-8-01, June 2008; "Vapor Intrusion Technical Guidance", New Jersey Department of Environmental Protection (NJDEP) Site Remediation Program, March 2013, Version 3.1 Method and laboratory SOP criteria, QAPP criteria and professional judgment	Angela Bown (or designee), CRA
Water/Solid	Dioxins and Furans (PCDDs & PCDFs)	Low, medium, high	USEPA National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs), EPA 540-R-05-001 September 2005; Method and laboratory SOP criteria, QAPP criteria and professional judgment	

QAPP Worksheet #37 - Usability Assessment

IDENTIFY THE PERSONNEL RESPONSIBLE FOR PERFORMING THE USABILITY ASSESSMENT:
Conestoga Rovers & Associates (CRA) will perform the usability assessment for analytical data. Angela Bown is responsible for data validation at CRA. The CRA Project Manager is responsible for verification of field activities. The CRA Project Manager and CRA QA Officer will determine if field and analytical data or datasets meet the requirements necessary for decision-making. The results of these measurements will be compared with the DQO requirements set forth in this QAPP. As data are evaluated, anomalies in the data or data gaps may become apparent to the data users. The DQOs will be satisfied if the data are sufficient (based on the quality and completeness of the data) to meet the project objectives. Data that do not meet the data users' needs (if any) will be identified and appropriately qualified in the project database so that the decision-makers are aware of the limitations.
SUMMARIZE THE USABILITY ASSESSMENT PROCESS AND ALL PROCEDURES, INCLUDING INTERIM STEPS AND ANY STATISTICS, EQUATIONS, AND COMPUTER ALGORITHMS THAT WILL BE USED:
The Data Reviewer will review field notes and field Chain-of-Custody Records to determine that procedures specified in the FSP have been followed. Validation of the analytical data will be performed by CRA chemistry staff at the direction of CRA's QA Officer based on the relevant and applicable evaluation criteria outlined in "USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review", EPA 540-R-8-01, June 2008, and "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review", EPA 540-R-10-011, January 2010 and the USEPA Analytical Services Branch National Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Data Review (EPA-540-R-05-001) and "Vapor Intrusion Technical Guidance", NJDEP Site Remediation Program, March 2013, Version 3.1. The elements reviewed, objectives established, evaluation approach, and actions taken will be as specified in these documents (referred to hereafter as NFGs) for the data quality assessment and validation. The specific criteria used will be as defined in the NFGs with the exception of the acceptance limits for the lab QC samples (surrogates, matrix spike/matrix spike duplicates, and laboratory control samples) which are either statistically determined by the laboratory as specified by the analytical methods and/or the specific control limits defined by the analytical methods and laboratory SOPs specified in this QAPP. The holding times, blanks, instrument performance checks, initial calibration, continuing calibration, internal standards, and serial dilutions requirements will be evaluated as specified in the NFGs where applicable, with select criteria for ICP-MS evaluation from "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review", EPA 540-R-10-011, January 2010. Data validation will determine whether the procedures specified in the FSP and in this QAPP were implemented, the DQOs specified in this QAPP were attained, the specified Quantitation Limits were achieved, and the sample holding times were met. An evaluation of data accuracy, precision, sensitivity, and completeness, based on method-specific criteria, will be performed according to the validation protocols presented above.

All forms will be checked. A representative portion (10 percent) of the sample raw data, including chromatograms, quantitation reports, data system printouts, and mass spectra will be randomly selected and reviewed. A portion (~10 percent) of calculations such as spike recoveries, calibration response factors, analyte quantitation, etc., will be randomly spot-checked.

The procedures used to evaluate data include the following:

- All technical holding times will be checked for inorganic and organic analyses.
- Instrument performance check sample results, and initial and continuing calibration results, will be evaluated.
- Data for all blanks, surrogate spikes, matrix spikes/matrix spike duplicates, laboratory control samples, cleanup standards, internaland external standards, target compound identification and quantitation, and system performance checks will be reviewed.
- Sample calculations will be checked.
- Field precision will be determined from blind field duplicate data.
- Completeness of the data package will be checked to determine if all samples and analyses required by the QAPP were processed, that the procedures specified in the QAPP were implemented, and that all deliverables specified in the QAPP are included.
- The Data Reviewer will identify any out-of-control data points and data omissions, and will interact with the laboratory to correct data deficiencies.
- Decisions to repeat sample collection and analyses may be made by the CRA Project Manager based on the extent of the deficiencies and their importance in the overall context of the project.
- The CRA QA Officer will assess the usability of results against the DQOs.

DESCRIBE THE EVALUATION PROCEDURES USED TO ASSESS OVERALL MEASUREMENT ERROR ASSOCIATED WITH THE PROJECT:

The data quality indicator (DQIs) used to evaluate conformance with the project DQOs are presented below.
DQIs are generally defined in terms of the following six parameters;

1. Representativeness
2. Comparability
3. Completeness
4. Precision
5. Accuracy
6. Sensitivity

Each parameter is defined below. Specific objectives for the site actions are presented in other sections of this QAPP, as referenced below:

Representativeness

Representativeness is the degree to which sampling data accurately and precisely represent site conditions, and is dependent on sampling and analytical variability and the variability of environmental media at the site. Actions have been designed to assess the presence of chemical constituents at the time of sampling. The QAPP presents the rationale for sample quantities and location. This QAPP presents field sampling and laboratory analytical methodologies. Use of the prescribed field and laboratory analytical methods with associated holding times and preservation requirements are intended to provide representative data.

Comparability

Comparability is the degree of confidence with which one data set can be compared with another. Comparability between phases of the actions (if additional phases are required) will be maintained through consistent use of the sampling and analytical methodologies set forth in the QAPP, the established QA/QC procedures, and the use of appropriately trained personnel.

Completeness

Completeness is defined as a measure of the amount of valid data obtained from an event and/or investigation compared to the total amount that was obtained. This will be determined upon final assessment of the analytical results. Completeness of a field or laboratory data set will be calculated by comparing the number of valid sample results generated with the total number of results generated.

$$\%\ Completeness = \frac{\text{Number of Valid Results}}{\text{Total Number of Results}} * 100$$

As a general guideline, overall project completeness is expected to be at least 90 percent. The assessment of completeness will require professional judgment to determine data usability for intended purposes.

Precision

Precision is a measure of the reproducibility of sample results. The goal is to maintain a level of analytical precision consistent with the objectives of the action. To maximize precision, sampling and analytical procedures will be followed. All work for the site actions will adhere to the established protocols presented in the QAPP. Checks for analytical precision will include the analysis of MS/MSDs, laboratory duplicates, and field duplicates. Checks for field measurement precision will include duplicate field measurements.

The precision of data will be measured by calculating the Relative Percent Difference (RPD) by the following equation:

$$RPD = \frac{(A - B)}{(A + B)/2} \times 100$$

Where:
A = analytical result from one of two duplicate measurements,
B = analytical result from the second measurement

Accuracy

Accuracy is a measure of how close a measured result is to the true value. Both field and analytical accuracy will be monitored through initial and continuing calibration of instruments. In addition, reference standards, MSs, blank spikes, and surrogate standards will be used to assess the accuracy of the analytical data.

Accuracy will be calculated in terms of percent recovery as follows:

$$\% = \frac{A - X}{B} \times 100$$

Where:
A = value measured in spiked sample or standard,
X = value measured in original sample,
B = true value of amount added to sample or true value of standard

Sensitivity
Sensitivity is a quantitative measurement to determine if the analytical laboratory's procedures/methodologies and their associated MDLs can satisfy the project requirements as they relate to the project action limits. MDLs are updated annually by the laboratory.

DESCRIBE THE DOCUMENTATION THAT WILL BE GENERATED DURING USABILITY ASSESSMENT AND HOW USABILITY ASSESSMENT RESULTS WILL BE PRESENTED SO THAT THEY IDENTIFY TRENDS, RELATIONSHIPS (CORRELATIONS), AND ANOMALIES:

The data validation report will address the following items:

- Overall quality and usability of the data
- Evaluation of QC data, including precision, accuracy, and completeness of the data
- Potential sample contamination due to blank contributions
- Assessment of laboratory and field records
- Actions regarding specific QC criteria exceedences

Laboratory-applied data qualifiers will be defined within the analytical data package received from the laboratory. The sample narrative will also detail quality control issues identified by the laboratory.

Data validation qualifiers that may be applied to the data include the following:

U	The analyte/compound was analyzed for, but not detected. The associated value is the compound's Limit of Quantitation.
UJ	The compound was not detected above the reported sample's Limit of Quantitation. However, the reported limit is approximate and may or may not represent the actual Limit of Quantitation.
J	The compound was positively identified; however, the associated numerical value is an estimated concentration only.
R	The sample results are rejected.

Appendix A

Laboratory Reference Data
TestAmerica – North Canton

alysis Group Descript	Method Description	Method Code
Waters	Volatile Organic Com	8260B

Analyte Description	CAS Number	RL	- Limit	RL	- Units	MDL	- Limit	MDL	- Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Acetone	67-64-1	10.0		µg/L		1.10		µg/L		43	136	%	30	%	33	145	%	30	%			%
Benzene	71-43-2	1.00		µg/L		0.130		µg/L		83	112	%	20	%	72	121	%	30	%			%
Dichlorobromomethan	75-27-4	1.00		µg/L		0.150		µg/L		72	121	%	30	%	67	120	%	30	%			%
Bromoform	75-25-2	1.00		µg/L		0.640		µg/L		40	131	%	30	%	32	128	%	30	%			%
Bromomethane	74-83-9	1.00		µg/L		0.410		µg/L		11	185	%	30	%	10	186	%	30	%			%
2-Butanone (MEK)	78-93-3	10.0		µg/L		0.570		µg/L		60	126	%	30	%	54	129	%	30	%			%
Carbon disulfide	75-15-0	1.00		µg/L		0.130		µg/L		62	142	%	30	%	57	147	%	30	%			%
Carbon tetrachloride	56-23-5	1.00		µg/L		0.130		µg/L		66	128	%	30	%	59	129	%	30	%			%
Chlorobenzene	108-90-7	1.00		µg/L		0.150		µg/L		85	110	%	30	%	80	110	%	30	%			%
Chloroethane	75-00-3	1.00		µg/L		0.290		µg/L		25	153	%	30	%	21	165	%	30	%			%
Chloroform	67-66-3	1.00		µg/L		0.160		µg/L		79	117	%	30	%	76	118	%	30	%			%
Chloromethane	74-87-3	1.00		µg/L		0.300		µg/L		44	126	%	30	%	33	132	%	30	%			%
1,1-Dichloroethane	75-34-3	1.00		µg/L		0.150		µg/L		82	115	%	30	%	79	116	%	30	%			%
1,2-Dichloroethane	107-06-2	1.00		µg/L		0.220		µg/L		71	127	%	30	%	68	129	%	30	%			%
1,1-Dichloroethene	75-35-4	1.00		µg/L		0.190		µg/L		78	131	%	30	%	74	135	%	30	%			%
1,2-Dichloropropane	78-87-5	1.00		µg/L		0.180		µg/L		81	115	%	30	%	78	115	%	30	%			%
cis-1,3-Dichloroprope	10061-01-5	1.00		µg/L		0.140		µg/L		61	115	%	30	%	51	110	%	30	%			%
trans-1,3-Dichloropro	10061-02-6	1.00		µg/L		0.190		µg/L		58	117	%	30	%	46	116	%	30	%			%
Ethylbenzene	100-41-4	1.00		µg/L		0.170		µg/L		83	112	%	30	%	75	116	%	30	%			%
2-Hexanone	591-78-6	10.0		µg/L		0.410		µg/L		55	133	%	30	%	47	139	%	30	%			%
Methylene Chloride	75-09-2	1.00		µg/L		0.330		µg/L		66	131	%	30	%	63	128	%	30	%			%
4-Methyl-2-pentanone	108-10-1	10.0		µg/L		0.320		µg/L		63	128	%	30	%	56	131	%	30	%			%
Styrene	100-42-5	1.00		µg/L		0.110		µg/L		79	114	%	30	%	71	117	%	30	%			%
1,1,2,2-Tetrachloroeth	79-34-5	1.00		µg/L		0.180		µg/L		68	118	%	30	%	63	122	%	30	%			%
Tetrachloroethene	127-18-4	1.00		µg/L		0.290		µg/L		79	114	%	30	%	70	117	%	30	%			%
Toluene	108-88-3	1.00		µg/L		0.130		µg/L		84	111	%	30	%	78	114	%	30	%			%
Trichloroethene	79-01-6	1.00		µg/L		0.170		µg/L		76	117	%	30	%	66	120	%	30	%			%
Vinyl chloride	75-01-4	1.00		µg/L		0.220		µg/L		53	127	%	30	%	49	130	%	30	%			%
Xylenes, Total	1330-20-7	2.00		µg/L		0.140		µg/L		83	112	%	30	%	76	116	%	30	%			%
1,1,1-Trichloroethane	71-55-6	1.00		µg/L		0.220		µg/L		74	118	%	30	%	68	121	%	30	%			%
1,1,2-Trichloroethane	79-00-5	1.00		µg/L		0.270		µg/L		80	112	%	30	%	75	115	%	30	%			%
Cyclohexane	110-82-7	1.00		µg/L		0.120		µg/L		54	121	%	30	%	49	123	%	30	%			%
1,2-Dibromo-3-Chloro	96-12-8	2.00		µg/L		0.670		µg/L		42	136	%	30	%	32	139	%	30	%			%
Ethylene Dibromide	106-93-4	1.00		µg/L		0.240		µg/L		79	113	%	30	%	74	113	%	30	%			%
Dichlorodifluorometha	75-71-8	1.00		µg/L		0.310		µg/L		19	129	%	30	%	17	128	%	30	%			%
cis-1,2-Dichloroethene	156-59-2	1.00		µg/L		0.170		µg/L		80	113	%	30	%	70	120	%	30	%			%
trans-1,2-Dichloroeth	156-60-5	1.00		µg/L		0.190		µg/L		83	117	%	30	%	80	119	%	30	%			%
Isopropylbenzene	98-82-8	1.00		µg/L		0.130		µg/L		75	114	%	30	%	68	116	%	30	%			%
Methyl acetate	79-20-9	10.0		µg/L		0.380		µg/L		58	131	%	30	%	47	130	%	30	%			%
Methyl tert-butyl ether	1634-04-4	1.00		µg/L		0.170		µg/L		52	144	%	30	%	46	144	%	30	%			%
1,1,2-Trichloro-1,2,2-t	76-13-1	1.00		µg/L		0.280		µg/L		74	151	%	30	%	70	152	%	30	%			%
1,2,4-Trichlorobenzen	120-82-1	1.00		µg/L		0.150		µg/L		48	135	%	30	%	38	138	%	30	%			%
1,2-Dichlorobenzene	95-50-1	1.00		µg/L		0.130		µg/L		81	110	%	30	%	75	111	%	30	%			%
1,3-Dichlorobenzene	541-73-1	1.00		µg/L		0.140		µg/L		80	110	%	30	%	73	110	%	30	%			%
1,4-Dichlorobenzene	106-46-7	1.00		µg/L		0.130		µg/L		82	110	%	30	%	75	110	%	30	%			%
Trichlorofluoromethan	75-69-4	1.00		µg/L		0.210		µg/L		49	157	%	30	%	46	157	%	30	%			%
Chlorodibromomethan	124-48-1	1.00		µg/L		0.180		µg/L		64	119	%	30	%	56	118	%	30	%			%
Methylcyclohexane	108-87-2	1.00		µg/L		0.130		µg/L		56	127	%	30	%	49	127	%	30	%			%
1,2-Dichloroethane-d4	17060-07-0			µg/L				µg/L				%	30	%			%	30	%	63	129	%
4-Bromofluorobenzen	460-00-4			µg/L				µg/L				%		%			%		%	66	117	%
Toluene-d8 (Surr)	2037-26-5			µg/L				µg/L				%	30	%			%	30	%	74	115	%
Dibromofluoromethan	1868-53-7			µg/L				µg/L				%	30	%			%	30	%	75	121	%
m-Xylene & p-Xylene	179601-23-1	2.00		µg/L		0.240		µg/L		83	113	%	30	%	75	117	%	30	%			%
o-Xylene	95-47-6	1.00		µg/L		0.140		µg/L		83	113	%	30	%	76	116	%	30	%			%
Naphthalene	91-20-3	1.00		µg/L		0.240		µg/L		32	141	%	30	%	15	158	%	30	%			%

Waters	Purge and Trap	5030B
Waters	Semivolatile Organic	8270C

Analyte Description	CAS Number	RL	- Limit	RL	- Units	MDL	- Limit	MDL	- Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	UREC - Recovery Lo	UREC - Recovery Hig	SUREC - Units
2-Fluorobiphenyl (Sur	321-60-8			µg/L				µg/L				%		%			%		%	20	110	%
2-Fluorophenol (Surr)	367-12-4			µg/L				µg/L				%		%			%		%	10	110	%
2,4,6-Tribromophenol	118-79-6			µg/L				µg/L				%		%			%		%	21	110	%
Nitrobenzene-d5 (Sur	4165-60-0			µg/L				µg/L				%		%			%		%	21	110	%
Phenol-d5 (Surr)	4165-62-2			µg/L				µg/L				%		%			%		%	21	110	%
Terphenyl-d14 (Surr)	1718-51-0			µg/L				µg/L				%		%			%		%	24	110	%
1,1'-Biphenyl	92-52-4	1.00		µg/L	0.130			µg/L		43	110	%	30	%	34	110	%	31	%			%
bis (2-chloroisopropyl	108-60-1	1.00		µg/L	0.400			µg/L		37	110	%	30	%	10	145	%	43	%			%
2,4,5-Trichlorophenol	95-95-4	5.00		µg/L	0.300			µg/L		48	110	%	30	%	36	110	%	60	%			%
2,4,6-Trichlorophenol	88-06-2	5.00		µg/L	0.240			µg/L		45	110	%	30	%	33	110	%	63	%			%
2,4-Dichlorophenol	120-83-2	2.00		µg/L	0.190			µg/L		41	110	%	30	%	28	110	%	69	%			%
2,4-Dimethylphenol	105-67-9	2.00		µg/L	0.250			µg/L		32	110	%	30	%	15	110	%	36	%			%
2,4-Dinitrophenol	51-28-5	5.00		µg/L	0.320			µg/L		10	110	%	30	%	10	124	%	70	%			%
2,4-Dinitrotoluene	121-14-2	5.00		µg/L	0.250			µg/L		53	110	%	30	%	37	110	%	56	%			%
2,6-Dinitrotoluene	606-20-2	5.00		µg/L	0.800			µg/L		54	110	%	30	%	38	110	%	54	%			%
2-Chloronaphthalene	91-58-7	1.00		µg/L	0.100			µg/L		43	110	%	30	%	28	110	%	37	%			%
2-Chlorophenol	95-57-8	1.00		µg/L	0.290			µg/L		29	110	%	30	%	20	110	%	70	%			%
2-Methylnaphthalene	91-57-6	0.200		µg/L	0.0904			µg/L		45	110	%	30	%	32	110	%	33	%			%
2-Methylphenol	95-48-7	1.00		µg/L	0.170			µg/L		42	110	%	30	%	27	110	%	42	%			%
2-Nitroaniline	88-74-4	2.00		µg/L	0.210			µg/L		54	110	%	30	%	38	110	%	32	%			%
2-Nitrophenol	88-75-5	2.00		µg/L	0.280			µg/L		40	110	%	30	%	26	110	%	64	%			%
3,3'-Dichlorobenzidine	91-94-1	5.00		µg/L	0.370			µg/L		22	110	%	30	%	10	110	%	99	%			%
3-Nitroaniline	99-09-2	2.00		µg/L	0.280			µg/L		53	110	%	30	%	22	110	%	69	%			%
4,6-Dinitro-2-methylpl	534-52-1	5.00		µg/L	2.40			µg/L		31	110	%	30	%	10	110	%	93	%			%
4-Bromophenyl phen	101-55-3	2.00		µg/L	0.220			µg/L		45	110	%	30	%	26	110	%	35	%			%
4-Chloro-3-methylphe	59-50-7	2.00		µg/L	0.210			µg/L		52	110	%	30	%	38	110	%	35	%			%
4-Chloroaniline	106-47-8	2.00		µg/L	0.210			µg/L		44	110	%	30	%	15	110	%	73	%			%
4-Chlorophenyl phen	7005-72-3	2.00		µg/L	0.300			µg/L		47	110	%	30	%	30	110	%	36	%			%
4-Nitroaniline	100-01-6	2.00		µg/L	0.220			µg/L		54	110	%	30	%	18	110	%	60	%			%
4-Nitrophenol	100-02-7	5.00		µg/L	0.290			µg/L		33	112	%	30	%	16	111	%	65	%			%
Acenaphthene	83-32-9	0.200		µg/L	0.0442			µg/L		47	110	%	30	%	35	110	%	30	%			%
Acenaphthylene	208-96-8	0.200		µg/L	0.0481			µg/L		49	110	%	30	%	33	110	%	30	%			%
Acetophenone	98-86-2	1.00		µg/L	0.340			µg/L		46	110	%	30	%	10	155	%	31	%			%
Anthracene	120-12-7	0.200		µg/L	0.0879			µg/L		52	110	%	30	%	26	110	%	37	%			%
Atrazine	1912-24-9	1.00		µg/L	0.340			µg/L		66	126	%	30	%	40	124	%	30	%			%
Benzaldehyde	100-52-7	1.00		µg/L	0.390			µg/L		38	110	%	30	%	24	110	%	34	%			%
Benzo[a]anthracene	56-55-3	0.200		µg/L	0.0295			µg/L		52	110	%	30	%	16	110	%	30	%			%
Benzo[a]pyrene	50-32-8	0.200		µg/L	0.0514			µg/L		44	110	%	30	%	10	110	%	60	%			%
Benzo[b]fluoranthene	205-99-2	0.200		µg/L	0.0394			µg/L		48	110	%	30	%	10	110	%	45	%			%
Benzo[g,h,i]perylene	191-24-2	0.200		µg/L	0.0464			µg/L		50	110	%	30	%	10	110	%	60	%			%
Benzo[k]fluoranthene	207-08-9	0.200		µg/L	0.0447			µg/L		49	110	%	30	%	10	110	%	48	%			%
Bis(2-chloroethoxy)me	111-91-1	1.00		µg/L	0.320			µg/L		43	110	%	30	%	27	110	%	33	%			%
Bis(2-chloroethyl)ethe	111-44-4	1.00		µg/L	0.100			µg/L		40	110	%	30	%	24	110	%	42	%			%
Bis(2-ethylhexyl) phth	117-81-7	2.00		µg/L	0.220			µg/L		39	116	%	30	%	10	112	%	71	%			%
Butyl benzyl phthalate	85-68-7	2.00		µg/L	0.260			µg/L		55	110	%	30	%	31	110	%	37	%			%
Caprolactam	105-60-2	5.00		µg/L	0.200			µg/L		45	111	%	30	%	10	199	%	99	%			%
Carbazole	86-74-8	1.00		µg/L	0.280			µg/L		55	110	%	30	%	28	110	%	30	%			%
Chrysene	218-01-9	0.200		µg/L	0.0502			µg/L		55	110	%	30	%	17	110	%	30	%			%
Dibenz(a,h)anthracen	53-70-3	0.200		µg/L	0.0446			µg/L		49	110	%	30	%	10	111	%	63	%			%
Dibenzofuran	132-64-9	1.00		µg/L	0.0200			µg/L		51	110	%	30	%	36	110	%	30	%			%
Diethyl phthalate	84-66-2	2.00		µg/L	0.600			µg/L		58	110	%	30	%	42	110	%	30	%			%
Dimethyl phthalate	131-11-3	2.00		µg/L	0.290			µg/L		57	110	%	30	%	42	110	%	30	%			%
Di-n-butyl phthalate	84-74-2	2.00		µg/L	0.670			µg/L		67	110	%	30	%	35	110	%	37	%			%
Di-n-octyl phthalate	117-84-0	2.00		µg/L	0.230			µg/L		40	110	%	30	%	10	118	%	92	%			%
Fluoranthene	206-44-0	0.200		µg/L	0.0446			µg/L		54	110	%	30	%	31	110	%	30	%			%
Fluorene	86-73-7	0.200		µg/L	0.0405			µg/L		52	110	%	30	%	36	110	%	30	%			%

Hexachlorobenzene	118-74-1	0.200	µg/L	0.0852	µg/L	50	110	%	30	%	23	110	%	30	%			%
Hexachlorobutadiene	87-68-3	1.00	µg/L	0.270	µg/L	33	110	%	30	%	15	110	%	49	%			%
Hexachlorocyclopenta	77-47-4	10.0	µg/L	0.240	µg/L	4	110	%	30	%	4	110	%	99	%			%
Hexachloroethane	67-72-1	1.00	µg/L	0.190	µg/L	35	110	%	30	%	10	122	%	44	%			%
Indeno[1,2,3-cd]pyren	193-39-5	0.200	µg/L	0.0433	µg/L	50	110	%	30	%	10	110	%	58	%			%
Isophorone	78-59-1	1.00	µg/L	0.270	µg/L	49	110	%	30	%	33	110	%	31	%			%
Naphthalene	91-20-3	0.200	µg/L	0.0627	µg/L	44	110	%	30	%	28	110	%	80	%			%
Nitrobenzene	98-95-3	1.00	µg/L	0.0400	µg/L	42	110	%	30	%	15	110	%	34	%			%
N-Nitrosodi-n-propyla	621-64-7	1.00	µg/L	0.240	µg/L	47	110	%	30	%	32	110	%	32	%			%
N-Nitrosodiphenylami	86-30-6	1.00	µg/L	0.310	µg/L	50	110	%	30	%	10	110	%	38	%			%
Pentachlorophenol	87-86-5	5.00	µg/L	0.270	µg/L	18	110	%	30	%	10	123	%	76	%			%
Phenol	108-95-2	1.00	µg/L	0.600	µg/L	33	110	%	30	%	25	110	%	74	%			%
Phenanthrene	85-01-8	0.200	µg/L	0.0619	µg/L	53	110	%	30	%	34	110	%	30	%			%
Pyrene	129-00-0	0.200	µg/L	0.0420	µg/L	52	110	%	30	%	32	110	%	30	%			%
3 & 4 Methylphenol	15831-10-4	2.00	µg/L	0.800	µg/L	44	110	%	30	%	31	110	%	42	%			%

Waters	Liquid-Liquid Extractio 3520C
Waters	Polychlorinated Biphe 8082

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Aroclor-1016	12674-11-2	0.500	µg/L	0.170	µg/L	66	120	%	30	%	67	120	%	30	%			%
Aroclor-1221	11104-28-2	0.500	µg/L	0.130	µg/L			%		%			%		%			%
Aroclor-1232	11141-16-5	0.500	µg/L	0.160	µg/L			%		%			%		%			%
Aroclor-1242	53469-21-9	0.500	µg/L	0.220	µg/L			%		%			%		%			%
Aroclor-1248	12672-29-6	0.500	µg/L	0.100	µg/L			%		%			%		%			%
Aroclor-1254	11097-69-1	0.500	µg/L	0.160	µg/L			%		%			%		%			%
Aroclor-1260	11096-82-5	0.500	µg/L	0.170	µg/L	55	120	%	30	%	31	120	%	30	%			%
Tetrachloro-m-xylene	877-09-8		µg/L		µg/L			%		%			%		%	23	136	%
DCB Decachlorobiphe	2051-24-3		µg/L		µg/L			%		%			%		%	10	130	%

Waters	Liquid-Liquid Extractio 3520C
Waters	Mercury (CVAA) 7470A

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Mercury	7439-97-6	0.200	µg/L	0.120	µg/L	81	123	%	20	%	69	134	%	20	%			

Waters	Preparation, Mercury 7470A_Prep
Waters	Metals (ICP) 6010B

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Aluminum	7429-90-5	200	µg/L	97.0	µg/L	80	120	%	20	%	75	125	%	20	%			
Antimony	7440-36-0	10.0	µg/L	1.80	µg/L	80	120	%	20	%	75	125	%	20	%			
Barium	7440-39-3	200	µg/L	0.670	µg/L	80	120	%	20	%	75	125	%	20	%			
Beryllium	7440-41-7	5.00	µg/L	0.460	µg/L	80	120	%	20	%	75	125	%	20	%			
Calcium	7440-70-2	5000	µg/L	130	µg/L	80	120	%	20	%	75	125	%	20	%			
Cadmium	7440-43-9	2.00	µg/L	0.660	µg/L	80	120	%	20	%	75	125	%	20	%			
Cobalt	7440-48-4	7.00	µg/L	1.70	µg/L	80	120	%	20	%	75	125	%	20	%			
Chromium	7440-47-3	5.00	µg/L	2.20	µg/L	80	120	%	20	%	75	125	%	20	%			
Copper	7440-50-8	25.0	µg/L	4.50	µg/L	80	120	%	20	%	75	125	%	20	%			
Iron	7439-89-6	100	µg/L	81.0	µg/L	80	120	%	20	%	75	125	%	20	%			
Potassium	7440-09-7	5000	µg/L	72.0	µg/L	80	120	%	20	%	75	125	%	20	%			
Magnesium	7439-95-4	5000	µg/L	34.0	µg/L	80	120	%	20	%	75	125	%	20	%			
Manganese	7439-96-5	15.0	µg/L	0.410	µg/L	80	120	%	20	%	75	125	%	20	%			
Silver	7440-22-4	5.00	µg/L	2.20	µg/L	80	120	%	20	%	75	125	%	20	%			
Sodium	7440-23-5	5000	µg/L	590	µg/L	80	120	%	20	%	75	125	%	20	%			
Nickel	7440-02-0	40.0	µg/L	3.20	µg/L	80	120	%	20	%	75	125	%	20	%			
Vanadium	7440-62-2	7.00	µg/L	0.640	µg/L	80	120	%	20	%	75	125	%	20	%			
Zinc	7440-66-6	20	µg/L	5.00	µg/L	80	120	%	20	%	75	125	%	20	%			
Arsenic	7440-38-2	10.0	µg/L	3.20	µg/L	80	120	%	20	%	75	125	%	20	%			

Lead	7439-92-1	3.00	µg/L	1.90	µg/L	80	120	%	20	%	80	75	125	%	20	%	
Selenium	7782-49-2	5.00	µg/L	4.10	µg/L	80	120	%	20	%	80	75	125	%	20	%	
Thallium	7440-28-0	10.0	µg/L	4.70	µg/L	80	120	%	20	%	80	75	125	%	20	%	

Waters	Preparation, Total Re
Waters	Diesel Range Organic

3005A	
8015B_DRO	

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
C10-C20	STL00115	500	µg/L	238	µg/L	40	124	%	30	%	27	120	%	31	%			%
C20-C34	STL00272	500	µg/L	238	µg/L	40	124	%	30	%	27	120	%	31	%			%
n-Nonane	111-84-2	500	µg/L		µg/L			%		%			%		%	20	120	%
Diesel Range Organic	STL00019	500	µg/L	238	µg/L	40	124	%	30	%	27	120	%	31	%			%

Waters	Liquid-Liquid Extractio
Waters	Gasoline Range Orga

3520C	
8015B_GRO	

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Gasoline Range Orga	8006-61-9	100	µg/L	25.0	µg/L	67	132	%	35	%	63	120	%	11	%			%
Trifluorotoluene (Surr	98-08-8		µg/L		µg/L			%		%			%		%	40	133	%

Waters	Purge and Trap
Waters	Organochlorine Pestic

5030B	
8081A	

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
4,4'-DDD	72-54-8	0.0500	µg/L	0.00960	µg/L	61	160	%	35	%	56	155	%	19	%			%
4,4'-DDE	72-55-9	0.0500	µg/L	0.00970	µg/L	50	160	%	35	%	47	137	%	35	%			%
4,4'-DDT	50-29-3	0.0500	µg/L	0.0160	µg/L	43	158	%	35	%	37	134	%	35	%			%
Aldrin	309-00-2	0.0500	µg/L	0.00820	µg/L	40	155	%	35	%	51	120	%	27	%			%
alpha-BHC	319-84-6	0.0500	µg/L	0.00700	µg/L	62	160	%	35	%	53	160	%	35	%			%
alpha-Chlordane	5103-71-9	0.0500	µg/L	0.0140	µg/L	44	160	%	35	%	42	153	%	35	%			%
beta-BHC	319-85-7	0.0500	µg/L	0.00840	µg/L	60	160	%	35	%	42	160	%	35	%			%
DCB Decachlorobiphe	2051-24-3		µg/L		µg/L			%		%			%		%	30	121	%
delta-BHC	319-86-8	0.0500	µg/L	0.00870	µg/L	55	167	%	35	%	49	160	%	35	%			%
Dieldrin	60-57-1	0.0500	µg/L	0.00750	µg/L	62	160	%	35	%	48	160	%	23	%			%
Endosulfan I	959-98-8	0.0500	µg/L	0.0130	µg/L	58	154	%	35	%	29	159	%	18	%			%
Endosulfan II	33213-65-9	0.0500	µg/L	0.0120	µg/L	56	145	%	35	%	35	142	%	26	%			%
Endosulfan sulfate	1031-07-8	0.0500	µg/L	0.0110	µg/L	64	151	%	35	%	47	150	%	32	%			%
Endrin	72-20-8	0.0500	µg/L	0.0110	µg/L	59	156	%	35	%	50	149	%	26	%			%
Endrin aldehyde	7421-93-4	0.0500	µg/L	0.0110	µg/L	58	136	%	35	%	42	149	%	31	%			%
Endrin ketone	53494-70-5	0.0500	µg/L	0.00780	µg/L	51	138	%	35	%	39	138	%	35	%			%
gamma-BHC (Lindane	58-89-9	0.0500	µg/L	0.00640	µg/L	65	158	%	35	%	53	160	%	31	%			%
gamma-Chlordane	5103-74-2	0.0500	µg/L	0.0120	µg/L	58	160	%	35	%	45	153	%	26	%			%
Heptachlor	76-44-8	0.0500	µg/L	0.00800	µg/L	40	143	%	35	%	36	128	%	35	%			%
Heptachlor epoxide	1024-57-3	0.0500	µg/L	0.00710	µg/L	61	160	%	35	%	35	160	%	24	%			%
Methoxychlor	72-43-5	0.100	µg/L	0.0320	µg/L	44	144	%	35	%	35	141	%	35	%			%
Toxaphene	8001-35-2	2.00	µg/L	0.320	µg/L			%	35	%	10	160	%	35	%			%
Tetrachloro-m-xylene	877-09-8		µg/L		µg/L			%		%			%		%	40	120	%
Toxaphene Peak 1	STL00100	2.00	µg/L	0.320	µg/L			%		%			%		%			%
Toxaphene Peak 2	STL00109	2.00	µg/L	0.320	µg/L			%		%			%		%			%
Toxaphene Peak 3	STL00220	2.00	µg/L	0.320	µg/L			%		%			%		%			%
Toxaphene Peak 4	STL00083	2.00	µg/L	0.320	µg/L			%		%			%		%			%
Toxaphene Peak 5	STL00051	2.00	µg/L	0.320	ug/L			%		%			%		%			%

Waters	Liquid-Liquid Extractio
Waters	Herbicides (GC)

3520C	
8151A	

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
2,4-D	94-75-7	4.00	µg/L	0.410	µg/L	63	141	%	35	%	71	133	%	21	%			%
2,4,5-T	93-76-5	1.00	µg/L	0.300	µg/L	57	146	%	35	%	74	129	%	17	%			%
Silvex (2,4,5-TP)	93-72-1	1.00	µg/L	0.200	µg/L	60	134	%	35	%	75	125	%	18	%			%
2,4-Dichlorophenylace	19719-28-9		µg/L		µg/L			%		%			%		%	52	123	%

Waters	Extraction (Herbicides
Waters	Dioxins and Furans (H

8151A_AP
8290

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
2,3,7,8-TCDD	1746-01-6	10.0	pg/L			72	144	%	20	%	72	144	%	20	%			
2,3,7,8-TCDF	51207-31-9	10.0	pg/L			73	150	%	20	%	73	150	%	20	%			
1,2,3,7,8-PeCDD	40321-76-4	50.0	pg/L			79	125	%	20	%	79	125	%	20	%			
1,2,3,7,8-PeCDF	57117-41-6	50.0	pg/L			79	137	%	20	%	79	137	%	20	%			
2,3,4,7,8-PeCDF	57117-31-4	50.0	pg/L			76	137	%	20	%	76	137	%	20	%			
1,2,3,4,7,8-HxCDD	39227-28-6	50.0	pg/L			65	144	%	20	%	65	144	%	20	%			
1,2,3,6,7,8-HxCDD	57653-85-7	50.0	pg/L			78	137	%	20	%	78	137	%	20	%			
1,2,3,7,8,9-HxCDD	19408-74-3	50.0	pg/L			74	142	%	20	%	74	142	%	20	%			
1,2,3,4,7,8-HxCDF	70648-26-9	50.0	pg/L			86	126	%	20	%	86	126	%	20	%			
1,2,3,6,7,8-HxCDF	57117-44-9	50.0	pg/L			79	137	%	20	%	79	137	%	20	%			
2,3,4,6,7,8-HxCDF	60851-34-5	50.0	pg/L			80	138	%	20	%	80	138	%	20	%			
1,2,3,7,8,9-HxCDF	72918-21-9	50.0	pg/L			72	145	%	20	%	72	145	%	20	%			
1,2,3,4,6,7,8-HpCDD	35822-46-9	50.0	pg/L			81	132	%	20	%	81	132	%	20	%			
1,2,3,4,6,7,8-HpCDF	67562-39-4	50.0	pg/L			81	135	%	20	%	81	135	%	20	%			
1,2,3,4,7,8,9-HpCDF	55673-89-7	50.0	pg/L			72	140	%	20	%	72	140	%	20	%			
OCDD	3268-87-9	100	pg/L			80	129	%	20	%	80	129	%	20	%			
OCDF	39001-02-0	100	pg/L			65	145	%	20	%	65	145	%	20	%			
Total TCDD	41903-57-5	10.0	pg/L					%		%			%		%			
Total TCDF	30402-14-3	10.0	pg/L					%		%			%		%			
Total PeCDD	36088-22-9	50.0	pg/L					%		%			%		%			
Total PeCDF	30402-15-4	50.0	pg/L					%		%			%		%			
Total HxCDD	34465-46-8	50.0	pg/L					%		%			%		%			
Total HxCDF	55684-94-1	50.0	pg/L					%		%			%		%			
Total HpCDD	37871-00-4	50.0	pg/L					%		%			%		%			
Total HpCDF	38998-75-3	50.0	pg/L					%		%			%		%			
13C-2,3,7,8-TCDD	76523-40-5		pg/L			40	135	%		%			%		%			
13C-2,3,7,8-TCDF	89059-46-1		pg/L			40	135	%		%			%		%			
13C-1,2,3,7,8-PeCDD	109719-79-1		pg/L			40	135	%		%			%		%			
13C-1,2,3,7,8-PeCDF	109719-77-9		pg/L			40	135	%		%			%		%			
13C-1,2,3,6,7,8-HxCD	109719-81-5		pg/L			40	135	%		%			%		%			
13C-1,2,3,4,7,8-HxCD	114423-98-2		pg/L			40	135	%		%			%		%			
13C-1,2,3,4,6,7,8-Hp	109719-83-7		pg/L			40	135	%		%			%		%			
13C-1,2,3,4,6,7,8-Hp	109719-84-8		pg/L			40	135	%		%			%		%			
13C-OCDD	114423-97-1		pg/L			40	135	%		%			%		%			

Waters	Separatory Funnel (Li
Waters	Cyanide, Total and/or

8290_P_Sep
9012A

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Cyanide, Total	57-12-5	0.0100	mg/L	0.00320	mg/L	69	118	%	20	%	42	140	%	20	%			

Waters	Cyanide, Total and/or
Waters	Alkalinity

9012A_Prep
2320B

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Alkalinity	STL00171	5.00	mg/L	1.90	mg/L	90	127	%	20	%	10	160	%	24	%			

Waters	Anions, Ion Chromato

300.0_28D

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Chloride	16887-00-6	1.00	mg/L	0.100	mg/L	90	110	%	20	%	80	120	%	20	%			
Sulfate	14808-79-8	1.00	mg/L	0.120	mg/L	90	110	%	20	%	80	120	%	20	%			

Waters	Organic Carbon, Diss

9060_Diss

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Dissolved Organic Ca	7440-44-0	1.00	mg/L	0.240	mg/L	88	115	%	20	%	72	136	%	20	%			

Waters	Sample Filtration, Fiel
Waters	Hardness, Total

FIELD_FLTRD
2340C

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Hardness as calcium	STL00009	5.00	mg/L	3.10	mg/L	88	110	%	20	%	87	114	%	20	%			

Waters	Anions, Ion Chromato

300

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Nitrite as N	14797-65-0	0.100	mg/L	0.0120	mg/L	90	110	%	20	%	80	120	%	20	%			
Nitrate as N	14797-55-8	0.100	mg/L	0.0230	mg/L	90	110	%	20	%	80	120	%	20	%			

Waters	Sulfide, Acid soluble a

9034_Calc

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Sulfide	18496-25-8	3.00	mg/L	0.940	mg/L	70	130	%	20	%	27	124	%	20	%			

Waters	Sulfide, Distillation (Ac
Waters	Dissolved Gases (GC

9030B

RSK_175

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
1,1,1-Trifluoroethane	420-46-2		µg/L		µg/L			%		%			%		%	66	132	%
Methane	74-82-8	0.500	µg/L	0.0700	µg/L	76	120	%	30	%	34	153	%	22	%			%
Ethane	74-84-0	0.500	µg/L	0.190	µg/L	80	120	%	30	%	61	120	%	21	%			%
Ethylene	74-85-1	0.500	µg/L	0.180	µg/L	81	120	%	30	%	60	120	%	17	%			%

Soils	Percent Moisture

Moisture

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Percent Solids	STL00234	0.100	%															
Percent Moisture	STL00177	0.100	%															

Soils	Polychlorinated Biphe

8082

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Aroclor-1016	12674-11-2	33.0	µg/Kg	21.0	µg/Kg	62	120	%	30	%	22	157	%	30	%			%
Aroclor-1221	11104-28-2	33.0	µg/Kg	16.0	µg/Kg			%		%			%		%			%
Aroclor-1232	11141-16-5	33.0	µg/Kg	14.0	µg/Kg			%		%			%		%			%
Aroclor-1242	53469-21-9	33.0	µg/Kg	13.0	µg/Kg			%		%			%		%			%
Aroclor-1248	12672-29-6	33.0	µg/Kg	17.0	µg/Kg			%		%			%		%			%
Aroclor-1254	11097-69-1	33.0	µg/Kg	17.0	µg/Kg			%		%			%		%			%
Aroclor-1260	11096-82-5	33.0	µg/Kg	17.0	µg/Kg	56	122	%	30	%	13	161	%	30	%			%
Tetrachloro-m-xylene	877-09-8		µg/Kg		µg/Kg			%		%			%		%	29	151	%
DCB Decachlorobiphe	2051-24-3		µg/Kg		µg/Kg			%		%			%		%	14	163	%

Soils	Soxhlet Extraction
Soils	Semivolatile Organic

3540C

8270C

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
2-Fluorobiphenyl (Sur	321-60-8		µg/Kg		µg/Kg			%		%			%		%	24	110	%
2-Fluorophenol (Surr)	367-12-4		µg/Kg		µg/Kg			%		%			%		%	24	110	%
2,4,6-Tribromophenol	118-79-6		µg/Kg		µg/Kg			%		%			%		%	10	110	%
Nitrobenzene-d5 (Sur	4165-60-0		µg/Kg		µg/Kg			%		%			%		%	20	110	%
Phenol-d5 (Surr)	4165-62-2		µg/Kg		µg/Kg			%		%			%		%	26	110	%
Terphenyl-d14 (Surr)	1718-51-0		µg/Kg		µg/Kg			%		%			%		%	36	110	%
1,1'-Biphenyl	92-52-4	50.0	µg/Kg	3.50	µg/Kg	35	110	%	30	%	32	110	%	32	%			%
bis (2-chloroisopropyl	108-60-1	100	µg/Kg	9.50	µg/Kg	29	110	%	30	%	11	110	%	42	%			%
2,4,5-Trichlorophenol	95-95-4	150	µg/Kg	25.0	µg/Kg	25	110	%	30	%	10	117	%	99	%			%
2,4,6-Trichlorophenol	88-06-2	150	µg/Kg	8.90	µg/Kg	12	110	%	30	%	10	110	%	38	%			%
2,4-Dichlorophenol	120-83-2	150	µg/Kg	20.0	µg/Kg	39	110	%	30	%	10	110	%	34	%			%
2,4-Dimethylphenol	105-67-9	150	µg/Kg	20.0	µg/Kg	29	110	%	30	%	10	110	%	31	%			%
2,4-Dinitrophenol	51-28-5	330	µg/Kg	21.0	µg/Kg	10	110	%	30	%	10	110	%	99	%			%
2,4-Dinitrotoluene	121-14-2	200	µg/Kg	17.0	µg/Kg	48	110	%	30	%	32	110	%	30	%			%
2,6-Dinitrotoluene	606-20-2	200	µg/Kg	21.0	µg/Kg	45	110	%	30	%	35	110	%	30	%			%
2-Chloronaphthalene	91-58-7	50.0	µg/Kg	0.450	µg/Kg	32	110	%	30	%	28	110	%	30	%			%
2-Chlorophenol	95-57-8	50.0	µg/Kg	8.20	µg/Kg	37	110	%	30	%	10	110	%	47	%			%

2-Methylnaphthalene	91-57-6	6.67	µg/Kg	0.500	µg/Kg	36	110	%	30	%	10	133	%	42	%			%
2-Methylphenol	95-48-7	200	µg/Kg	11.0	µg/Kg	41	110	%	30	%	24	110	%	51	%			%
2-Nitroaniline	88-74-4	200	µg/Kg	9.10	µg/Kg	45	110	%	30	%	39	110	%	31	%			%
2-Nitrophenol	88-75-5	50.0	µg/Kg	8.30	µg/Kg	34	110	%	30	%	10	110	%	49	%			%
3,3'-Dichlorobenzidine	91-94-1	100	µg/Kg	18.0	µg/Kg	28	110	%	30	%	10	110	%	56	%			%
3-Nitroaniline	99-09-2	200	µg/Kg	16.0	µg/Kg	44	110	%	30	%	10	110	%	30	%			%
4,6-Dinitro-2-methylph	534-52-1	150	µg/Kg	9.20	µg/Kg	10	110	%	30	%	10	110	%	55	%			%
4-Bromophenyl phenyl	101-55-3	50.0	µg/Kg	13.0	µg/Kg	39	110	%	30	%	33	110	%	30	%			%
4-Chloro-3-methylphe	59-50-7	150	µg/Kg	21.0	µg/Kg	48	110	%	30	%	25	110	%	54	%			%
4-Chloroaniline	106-47-8	150	µg/Kg	17.0	µg/Kg	30	110	%	30	%	10	110	%	36	%			%
4-Chlorophenyl phenyl	7005-72-3	50.0	µg/Kg	13.0	µg/Kg	40	110	%	30	%	32	110	%	30	%			%
4-Nitroaniline	100-01-6	200	µg/Kg	26.0	µg/Kg	48	110	%	30	%	10	110	%	48	%			%
4-Nitrophenol	100-02-7	330	µg/Kg	17.0	µg/Kg	28	110	%	30	%	10	113	%	49	%			%
Acenaphthene	83-32-9	6.67	µg/Kg	0.760	µg/Kg	38	110	%	30	%	22	110	%	99	%			%
Acenaphthylene	208-96-8	6.67	µg/Kg	0.350	µg/Kg	40	110	%	30	%	24	110	%	99	%			%
Acetophenone	98-86-2	100	µg/Kg	9.20	µg/Kg	40	110	%	30	%	31	110	%	43	%			%
Anthracene	120-12-7	6.67	µg/Kg	0.780	µg/Kg	48	110	%	30	%	20	110	%	99	%			%
Atrazine	1912-24-9	200	µg/Kg	9.10	µg/Kg	66	127	%	30	%	45	118	%	30	%			%
Benzaldehyde	100-52-7	100	µg/Kg	12.0	µg/Kg	32	110	%	30	%	23	110	%	42	%			%
Benzo[a]anthracene	56-55-3	6.67	µg/Kg	0.630	µg/Kg	50	110	%	30	%	10	122	%	99	%			%
Benzo[a]pyrene	50-32-8	6.67	µg/Kg	0.640	µg/Kg	44	110	%	30	%	10	110	%	99	%			%
Benzo[b]fluoranthene	205-99-2	6.67	µg/Kg	0.590	µg/Kg	43	110	%	30	%	12	118	%	99	%			%
Benzo[g,h,i]perylene	191-24-2	6.67	µg/Kg	0.350	µg/Kg	51	110	%	30	%	10	117	%	99	%			%
Benzo[k]fluoranthene	207-08-9	6.67	µg/Kg	0.680	µg/Kg	38	105	%	30	%	10	121	%	99	%			%
Bis(2-chloroethoxy)me	111-91-1	100	µg/Kg	22.0	µg/Kg	32	110	%	30	%	26	110	%	37	%			%
Bis(2-chloroethyl)ethe	111-44-4	100	µg/Kg	2.00	µg/Kg	34	110	%	30	%	21	110	%	55	%			%
Bis(2-ethylhexyl) phth	117-81-7	70.0	µg/Kg	19.0	µg/Kg	50	110	%	30	%	40	110	%	30	%			%
Butyl benzyl phthalate	85-68-7	70.0	µg/Kg	10.0	µg/Kg	51	110	%	30	%	44	110	%	30	%			%
Caprolactam	105-60-2	330	µg/Kg	37.0	µg/Kg	44	114	%	30	%	10	134	%	32	%			%
Carbazole	86-74-8	50.0	µg/Kg	27.0	µg/Kg	50	110	%	30	%	34	110	%	30	%			%
Chrysene	218-01-9	6.67	µg/Kg	1.10	µg/Kg	50	110	%	30	%	10	125	%	99	%			%
Dibenz(a,h)anthracen	53-70-3	6.67	µg/Kg	0.660	µg/Kg	51	110	%	30	%	14	113	%	99	%			%
Dibenzofuran	132-64-9	50.0	µg/Kg	0.660	µg/Kg	43	110	%	30	%	29	110	%	30	%			%
Diethyl phthalate	84-66-2	70.0	µg/Kg	16.0	µg/Kg	52	110	%	30	%	42	110	%	30	%			%
Dimethyl phthalate	131-11-3	70.0	µg/Kg	17.0	µg/Kg	50	110	%	30	%	41	110	%	30	%			%
Di-n-butyl phthalate	84-74-2	70.0	µg/Kg	15.0	µg/Kg	51	110	%	30	%	43	110	%	30	%			%
Di-n-octyl phthalate	117-84-0	70.0	µg/Kg	7.90	µg/Kg	48	110	%	30	%	24	119	%	30	%			%
Fluoranthene	206-44-0	6.67	µg/Kg	0.550	µg/Kg	51	110	%	30	%	10	110	%	99	%			%
Fluorene	86-73-7	6.67	µg/Kg	0.530	µg/Kg	46	110	%	30	%	23	110	%	99	%			%
Hexachlorobenzene	118-74-1	6.67	µg/Kg	2.10	µg/Kg	43	110	%	30	%	34	110	%	30	%			%
Hexachlorobutadiene	87-68-3	50.0	µg/Kg	5.60	µg/Kg	29	110	%	30	%	25	110	%	34	%			%
Hexachlorocyclopenta	77-47-4	330	µg/Kg	8.10	µg/Kg	12	110	%	30	%	10	110	%	79	%			%
Hexachloroethane	67-72-1	50.0	µg/Kg	9.00	µg/Kg	30	110	%	30	%	12	110	%	50	%			%
Indeno[1,2,3-cd]pyren	193-39-5	6.67	µg/Kg	0.350	µg/Kg	50	110	%	30	%	10	114	%	99	%			%
Isophorone	78-59-1	50.0	µg/Kg	13.0	µg/Kg	36	110	%	30	%	29	110	%	38	%			%
Naphthalene	91-20-3	6.67	µg/Kg	0.820	µg/Kg	36	110	%	30	%	10	111	%	99	%			%
Nitrobenzene	98-95-3	100	µg/Kg	2.20	µg/Kg	32	110	%	30	%	23	110	%	41	%			%
N-Nitrosodi-n-propyla	621-64-7	50.0	µg/Kg	6.30	µg/Kg	38	110	%	30	%	26	110	%	42	%			%
N-Nitrosodiphenylami	86-30-6	50.0	µg/Kg	21.0	µg/Kg	46	110	%	30	%	22	110	%	30	%			%
Pentachlorophenol	87-86-5	150	µg/Kg	9.10	µg/Kg	10	110	%	30	%	10	110	%	50	%			%
Phenol	108-95-2	50.0	µg/Kg	7.30	µg/Kg	38	110	%	30	%	17	110	%	53	%			%
Phenanthrene	85-01-8	6.67	µg/Kg	0.730	µg/Kg	49	110	%	30	%	10	166	%	99	%			%
Pyrene	129-00-0	6.67	µg/Kg	0.440	µg/Kg	49	110	%	30	%	10	147	%	99	%			%
3 & 4 Methylphenol	15831-10-4	400	µg/Kg	20.0	µg/Kg	40	110	%	30	%	25	110	%	50	%			%

Soils	Soxhlet Extraction	3540C
Soils	Mercury (CVAA)	7471A

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	SREC - Recovery Lo	SREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	UREC - Recovery Lo	UREC - Recovery Hig	SUREC - Units
Mercury	7439-97-6	0.100	mg/Kg	0.0150	mg/Kg	73	121	%	20	%	11	192	%	20	%			

Soils	Preparation, Mercury	7471A_Prep
Soils	Metals (ICP)	6010B

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	UREC - Recovery Lo	UREC - Recovery Hig	SUREC - Units
Aluminum	7429-90-5	20.0	mg/Kg	9.60	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Antimony	7440-36-0	1.00	mg/Kg	0.390	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Barium	7440-39-3	20.0	mg/Kg	0.0710	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Beryllium	7440-41-7	0.500	mg/Kg	0.0430	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Calcium	7440-70-2	500	mg/Kg	16.0	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Cadmium	7440-43-9	0.200	mg/Kg	0.0360	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Cobalt	7440-48-4	5.00	mg/Kg	0.160	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Chromium	7440-47-3	0.500	mg/Kg	0.200	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Copper	7440-50-8	2.50	mg/Kg	0.740	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Iron	7439-89-6	10.0	mg/Kg	4.90	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Potassium	7440-09-7	500	mg/Kg	6.20	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Magnesium	7439-95-4	500	mg/Kg	5.10	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Manganese	7439-96-5	1.50	mg/Kg	0.0740	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Silver	7440-22-4	0.500	mg/Kg	0.100	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Sodium	7440-23-5	500	mg/Kg	66.0	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Nickel	7440-02-0	4.00	mg/Kg	0.270	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Vanadium	7440-62-2	5.00	mg/Kg	0.120	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Zinc	7440-66-6	2.00	mg/Kg	1.00	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Arsenic	7440-38-2	1.00	mg/Kg	0.300	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Lead	7439-92-1	0.300	mg/Kg	0.190	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Selenium	7782-49-2	0.500	mg/Kg	0.450	mg/Kg	80	120	%	20	%	75	125	%	20	%			
Thallium	7440-28-0	1.00	mg/Kg	0.550	mg/Kg	80	120	%	20	%	75	125	%	20	%			

Soils	Preparation, Metals	3050B
Soils	Volatile Organic Com	8260B

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	UREC - Recovery Lo	UREC - Recovery Hig	SUREC - Units
Acetone	67-64-1	1000	µg/Kg	170	µg/Kg	16	156	%	30	%	10	142	%	30	%	10	142	%
Benzene	71-43-2	250	µg/Kg	12.0	µg/Kg	70	117	%	30	%	10	199	%	30	%	10	199	%
Dichlorobromomethan	75-27-4	250	µg/Kg	9.90	µg/Kg	28	123	%	30	%	18	133	%	30	%	18	133	%
Bromoform	75-25-2	250	µg/Kg	19.0	µg/Kg	10	117	%	30	%	10	147	%	30	%	10	147	%
Bromomethane	74-83-9	250	µg/Kg	29.0	µg/Kg	10	114	%	30	%	10	151	%	30	%	10	151	%
2-Butanone (MEK)	78-93-3	1000	µg/Kg	43.0	µg/Kg	10	199	%	30	%	10	172	%	30	%	10	172	%
Carbon disulfide	75-15-0	250	µg/Kg	12.0	µg/Kg	10	132	%	30	%	10	155	%	30	%	10	155	%
Carbon tetrachloride	56-23-5	250	µg/Kg	6.40	µg/Kg	29	118	%	30	%	12	135	%	30	%	12	135	%
Chlorobenzene	108-90-7	250	µg/Kg	6.40	µg/Kg	71	116	%	30	%	47	118	%	30	%	47	118	%
Chloroethane	75-00-3	250	µg/Kg	61.0	µg/Kg	10	120	%	30	%	10	168	%	30	%	10	168	%
Chloroform	67-66-3	250	µg/Kg	8.80	µg/Kg	63	116	%	30	%	51	120	%	30	%	51	120	%
Chloromethane	74-87-3	250	µg/Kg	14.0	µg/Kg	25	110	%	30	%	16	115	%	30	%	16	115	%
1,1-Dichloroethane	75-34-3	250	µg/Kg	17.0	µg/Kg	63	117	%	30	%	18	160	%	30	%	18	160	%
1,2-Dichloroethane	107-06-2	250	µg/Kg	10.0	µg/Kg	68	119	%	30	%	25	150	%	30	%	25	150	%
1,1-Dichloroethene	75-35-4	250	µg/Kg	18.0	µg/Kg	44	143	%	30	%	10	179	%	30	%	10	179	%
1,2-Dichloropropane	78-87-5	250	µg/Kg	8.20	µg/Kg	73	113	%	30	%	58	118	%	30	%	58	118	%
cis-1,3-Dichloroprope	10061-01-5	250	µg/Kg	7.90	µg/Kg	25	120	%	30	%	19	121	%	30	%	19	121	%
trans-1,3-Dichloropro	10061-02-6	250	µg/Kg	20.0	µg/Kg	22	122	%	30	%	10	136	%	30	%	10	136	%
Ethylbenzene	100-41-4	250	µg/Kg	5.40	µg/Kg	66	119	%	30	%	27	143	%	30	%	27	143	%
2-Hexanone	591-78-6	1000	µg/Kg	20.0	µg/Kg	43	130	%	30	%	21	141	%	30	%	21	141	%
Methylene Chloride	75-09-2	250	µg/Kg	77.0	µg/Kg	27	172	%	30	%	10	148	%	30	%	10	148	%
4-Methyl-2-pentanone	108-10-1	1000	µg/Kg	48.0	µg/Kg	49	121	%	30	%	19	151	%	30	%	19	151	%
Styrene	100-42-5	250	µg/Kg	5.60	µg/Kg	60	120	%	30	%	31	137	%	30	%	31	137	%
1,1,2,2-Tetrachloroeth	79-34-5	250	µg/Kg	8.90	µg/Kg	54	121	%	30	%	16	158	%	30	%	16	158	%
Tetrachloroethene	127-18-4	250	µg/Kg	12.0	µg/Kg	58	131	%	30	%	19	153	%	30	%	19	153	%
Toluene	108-88-3	250	µg/Kg	17.0	µg/Kg	66	123	%	30	%	10	168	%	30	%	10	168	%
Trichloroethene	79-01-6	250	µg/Kg	9.70	µg/Kg	59	124	%	30	%	10	193	%	30	%	10	193	%
Vinyl chloride	75-01-4	250	µg/Kg	18.0	µg/Kg	33	110	%	30	%	15	123	%	30	%	15	123	%
Xylenes, Total	1330-20-7	500	µg/Kg	6.20	µg/Kg	68	119	%	30	%	16	150	%	30	%	16	150	%

1,1,1-Trichloroethane	71-55-6	250	µg/Kg	21.0	µg/Kg	38	122	%	30	%	10	159	%	30	%	10	159	%
1,1,2-Trichloroethane	79-00-5	250	µg/Kg	12.0	µg/Kg	74	114	%	30	%	34	152	%	30	%	34	152	%
Cyclohexane	110-82-7	500	µg/Kg	40.0	µg/Kg	40	120	%	30	%	10	154	%	30	%	10	154	%
1,2-Dibromo-3-Chloro	96-12-8	500	µg/Kg	50.0	µg/Kg	10	129	%	30	%	10	137	%	30	%			%
Ethylene Dibromide	106-93-4	250	µg/Kg	10.0	µg/Kg	47	123	%	30	%	32	127	%	30	%			%
Dichlorodifluorometha	75-71-8	250	µg/Kg	16.0	µg/Kg	10	110	%	30	%	10	113	%	30	%	10	113	%
cis-1,2-Dichloroethen	156-59-2	250	µg/Kg	6.90	µg/Kg	60	125	%	30	%	34	137	%	30	%	34	137	%
trans-1,2-Dichloroethe	156-60-5	250	µg/Kg	9.20	µg/Kg	58	121	%	30	%	40	126	%	30	%	40	126	%
Isopropylbenzene	98-82-8	250	µg/Kg	6.50	µg/Kg	61	123	%	30	%	39	126	%	30	%	39	126	%
Methyl acetate	79-20-9	500	µg/Kg	25.0	µg/Kg	44	173	%	30	%	10	175	%	30	%	10	175	%
Methyl tert-butyl ether	1634-04-4	250	µg/Kg	7.10	µg/Kg	34	157	%	30	%	26	159	%	30	%	26	159	%
1,1,2-Trichloro-1,2,2-t	76-13-1	250	µg/Kg	39.0	µg/Kg	48	151	%	30	%	23	168	%	30	%	23	168	%
1,2,4-Trichlorobenzen	120-82-1	250	µg/Kg	7.30	µg/Kg	41	135	%	30	%	10	136	%	30	%	10	136	%
1,2-Dichlorobenzene	95-50-1	250	µg/Kg	8.60	µg/Kg	68	118	%	30	%	27	126	%	30	%	27	126	%
1,3-Dichlorobenzene	541-73-1	250	µg/Kg	4.80	µg/Kg	66	121	%	30	%	29	124	%	30	%	29	124	%
1,4-Dichlorobenzene	106-46-7	250	µg/Kg	8.00	µg/Kg	65	119	%	30	%	30	123	%	30	%	30	123	%
Trichlorofluoromethan	75-69-4	250	µg/Kg	16.0	µg/Kg	17	145	%	30	%	10	157	%	30	%	10	157	%
Chlorodibromomethan	124-48-1	250	µg/Kg	12.0	µg/Kg	22	113	%	30	%	10	128	%	30	%	10	128	%
Methylcyclohexane	108-87-2	500	µg/Kg	12.0	µg/Kg	41	133	%	30	%	11	156	%	30	%	11	156	%
1,2-Dichloroethane-d4	17060-07-0	250	µg/Kg	0.0100	µg/Kg			%	30	%			%	30	%	39	128	%
4-Bromofluorobenzen	460-00-4	250	µg/Kg	0.0100	µg/Kg			%	30	%			%	30	%	26	141	%
Toluene-d8 (Surr)	2037-26-5	250	µg/Kg	0.0100	µg/Kg			%	30	%			%	30	%	33	134	%
Dibromofluoromethan	1868-53-7	250	µg/Kg	0.0100	µg/Kg			%	30	%			%	30	%	30	122	%
m-Xylene & p-Xylene	179601-23-1	250	µg/Kg	6.20	µg/Kg	67	118	%	30	%	14	151	%	30	%	14	151	%
o-Xylene	95-47-6	250	µg/Kg	8.50	µg/Kg	68	120	%	30	%	18	151	%	30	%	18	151	%

Soils	Closed System Purge	5035A_M
Soils	Volatile Organic Com	8260B

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Acetone	67-64-1	20.0	µg/Kg	6.30	µg/Kg	41	137	%	30	%	24	140	%	30	%	24	140	%
Benzene	71-43-2	5.00	µg/Kg	0.230	µg/Kg	79	112	%	30	%	53	118	%	30	%	53	118	%
Dichlorobromomethan	75-27-4	5.00	µg/Kg	0.280	µg/Kg	84	122	%	30	%	35	132	%	30	%	35	132	%
Bromoform	75-25-2	5.00	µg/Kg	0.330	µg/Kg	62	133	%	30	%	18	129	%	30	%	18	129	%
Bromomethane	74-83-9	5.00	µg/Kg	0.540	µg/Kg	42	136	%	30	%	33	130	%	30	%	33	130	%
2-Butanone (MEK)	78-93-3	20.0	µg/Kg	1.40	µg/Kg	52	131	%	30	%	30	143	%	30	%	30	143	%
Carbon disulfide	75-15-0	5.00	µg/Kg	0.440	µg/Kg	62	146	%	30	%	20	151	%	30	%	20	151	%
Carbon tetrachloride	56-23-5	5.00	µg/Kg	0.370	µg/Kg	71	129	%	30	%	32	137	%	30	%	32	137	%
Chlorobenzene	108-90-7	5.00	µg/Kg	0.330	µg/Kg	78	110	%	30	%	37	116	%	30	%	37	116	%
Chloroethane	75-00-3	5.00	µg/Kg	0.860	µg/Kg	58	117	%	30	%	45	118	%	30	%	45	118	%
Chloroform	67-66-3	5.00	µg/Kg	0.290	µg/Kg	77	114	%	30	%	53	119	%	30	%	53	119	%
Chloromethane	74-87-3	5.00	µg/Kg	0.410	µg/Kg	50	110	%	30	%	34	117	%	30	%	34	117	%
1,1-Dichloroethane	75-34-3	5.00	µg/Kg	0.360	µg/Kg	76	115	%	30	%	54	122	%	30	%	54	122	%
1,2-Dichloroethane	107-06-2	5.00	µg/Kg	0.340	µg/Kg	72	120	%	30	%	49	123	%	30	%	49	123	%
1,1-Dichloroethene	75-35-4	5.00	µg/Kg	0.520	µg/Kg	75	135	%	30	%	49	157	%	30	%	49	157	%
1,2-Dichloropropane	78-87-5	5.00	µg/Kg	0.690	µg/Kg	87	113	%	30	%	61	117	%	30	%	61	117	%
cis-1,3-Dichloroprope	10061-01-5	5.00	µg/Kg	0.340	µg/Kg	74	128	%	30	%	27	133	%	30	%	27	133	%
trans-1,3-Dichloropro	10061-02-6	5.00	µg/Kg	0.540	µg/Kg	73	131	%	30	%	28	137	%	30	%	28	137	%
Ethylbenzene	100-41-4	5.00	µg/Kg	0.260	µg/Kg	79	117	%	30	%	30	131	%	30	%	30	131	%
2-Hexanone	591-78-6	20.0	µg/Kg	0.630	µg/Kg	64	136	%	30	%	37	147	%	30	%	37	147	%
Methylene Chloride	75-09-2	5.00	µg/Kg	0.670	µg/Kg	75	118	%	30	%	54	115	%	30	%	54	115	%
4-Methyl-2-pentanone	108-10-1	20.0	µg/Kg	0.540	µg/Kg	67	135	%	30	%	43	147	%	30	%	43	147	%
Styrene	100-42-5	5.00	µg/Kg	0.150	µg/Kg	87	117	%	30	%	27	127	%	30	%	27	127	%
1,1,2,2-Tetrachloroeth	79-34-5	5.00	µg/Kg	0.340	µg/Kg	77	123	%	30	%	16	179	%	30	%	16	179	%
Tetrachloroethene	127-18-4	5.00	µg/Kg	0.520	µg/Kg	79	114	%	30	%	31	135	%	30	%	31	135	%
Toluene	108-88-3	5.00	µg/Kg	0.270	µg/Kg	75	111	%	30	%	39	129	%	30	%	39	129	%
Trichloroethene	79-01-6	5.00	µg/Kg	0.420	µg/Kg	79	113	%	30	%	10	177	%	30	%	10	177	%
Vinyl chloride	75-01-4	5.00	µg/Kg	0.390	µg/Kg	67	114	%	30	%	42	117	%	30	%	42	117	%
Xylenes, Total	1330-20-7	10.0	µg/Kg	0.350	µg/Kg	80	118	%	30	%	30	131	%	30	%	30	131	%
1,1,1-Trichloroethane	71-55-6	5.00	µg/Kg	0.560	µg/Kg	77	126	%	30	%	51	128	%	30	%	51	128	%

1,1,2-Trichloroethane	79-00-5	5.00	µg/Kg	0.390	µg/Kg	83	112	%	30	%	10	166	%	30	%	10	166	%	
Cyclohexane	110-82-7	10.0	µg/Kg	0.330	µg/Kg	66	110	%	30	%	28	118	%	30	%	28	118	%	
1,2-Dibromo-3-Chloro	96-12-8	10.0	µg/Kg	1.30	µg/Kg	61	132	%	30	%	10	153	%	30	%			%	
Ethylene Dibromide	106-93-4	5.00	µg/Kg	0.500	µg/Kg	83	117	%	30	%	45	127	%	30	%			%	
Dichlorodifluorometha	75-71-8	5.00	µg/Kg	0.500	µg/Kg	26	113	%	30	%	17	115	%	30	%	17	115	%	
cis-1,2-Dichloroethene	156-59-2	5.00	µg/Kg	0.360	µg/Kg	76	113	%	30	%	50	119	%	30	%	50	119	%	
trans-1,2-Dichloroethe	156-60-5	5.00	µg/Kg	0.410	µg/Kg	78	117	%	30	%	50	123	%	30	%	50	123	%	
Isopropylbenzene	98-82-8	5.00	µg/Kg	0.160	µg/Kg	76	122	%	30	%	21	134	%	30	%	21	134	%	
Methyl acetate	79-20-9	10.0	µg/Kg	1.40	µg/Kg	57	130	%	30	%	33	165	%	30	%	33	165	%	
Methyl tert-butyl ether	1634-04-4	5.00	µg/Kg	0.430	µg/Kg	49	165	%	30	%	51	157	%	30	%	51	157	%	
1,1,2-Trichloro-1,2,2-t	76-13-1	5.00	µg/Kg	1.30	µg/Kg	82	138	%	30	%	50	147	%	30	%	50	147	%	
1,2,4-Trichlorobenzen	120-82-1	5.00	µg/Kg	0.270	µg/Kg	64	124	%	30	%	10	111	%	30	%	10	111	%	
1,2-Dichlorobenzene	95-50-1	5.00	µg/Kg	0.360	µg/Kg	76	110	%	30	%	17	122	%	30	%	17	122	%	
1,3-Dichlorobenzene	541-73-1	5.00	µg/Kg	0.350	µg/Kg	78	111	%	30	%	16	126	%	30	%	16	126	%	
1,4-Dichlorobenzene	106-46-7	5.00	µg/Kg	0.660	µg/Kg	75	110	%	30	%	15	121	%	30	%	15	121	%	
Trichlorofluoromethan	75-69-4	5.00	µg/Kg	0.340	µg/Kg	57	146	%	30	%	36	142	%	30	%	36	142	%	
Chlorodibromomethan	124-48-1	5.00	µg/Kg	0.550	µg/Kg	72	127	%	30	%	29	135	%	30	%	29	135	%	
Methylcyclohexane	108-87-2	10.0	µg/Kg	0.310	µg/Kg	70	126	%	30	%	20	132	%	30	%	20	132	%	
1,2-Dichloroethane-d4	17060-07-0		µg/Kg		µg/Kg			%	30	%			%	30	%	58	123	%	
4-Bromofluorobenzen	460-00-4		µg/Kg		µg/Kg			%		%			%		%	52	136	%	
Toluene-d8 (Surr)	2037-26-5		µg/Kg		µg/Kg			%	30	%			%	30	%	67	125	%	
Dibromofluoromethan	1868-53-7		µg/Kg		µg/Kg			%	30	%			%	30	%	37	132	%	
m-Xylene & p-Xylene	179601-23-1	10.0	µg/Kg	1.20	µg/Kg	80	117	%	30	%	29	131	%	30	%	29	131	%	
o-Xylene	95-47-6	5.00	µg/Kg	0.350	µg/Kg	80	120	%	30	%	29	134	%	30	%	29	134	%	

Soils	Closed System Purge
Soils	Cyanide, Total and/or

5035A_FW	
9012A	

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Cyanide, Total	57-12-5	0.500	mg/Kg	0.100	mg/Kg	68	123	%	20	%	50	134	%	20	%			

Soils	Cyanide, Total and/or
Soils	Sulfide, Acid soluble a

9012A_Prep	
9034_Calc	

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Sulfide	18496-25-8	30.0	mg/Kg	22.0	mg/Kg	70	130	%	20	%	10	154	%	20	%			

Soils	Sulfide, Distillation (Ad
Soils	pH

9030B	
9040C	

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Corrosivity	STL00179	0.100	SU			97	103	%	20	%								

Soils	Organochlorine Pestic

8081A	

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
4,4'-DDD	72-54-8	1.70	µg/Kg	0.620	µg/Kg	53	160	%	40	%	16	160	%	40	%			%
4,4'-DDE	72-55-9	1.70	µg/Kg	0.390	µg/Kg	46	143	%	40	%	37	150	%	40	%			%
4,4'-DDT	50-29-3	1.70	µg/Kg	0.630	µg/Kg	40	157	%	40	%	24	160	%	40	%			%
Aldrin	309-00-2	1.70	µg/Kg	1.20	µg/Kg	40	145	%	40	%	41	137	%	40	%			%
alpha-BHC	319-84-6	1.70	µg/Kg	0.730	µg/Kg	50	153	%	40	%	22	160	%	40	%			%
alpha-Chlordane	5103-71-9	1.70	µg/Kg	0.940	µg/Kg	42	150	%	40	%	38	145	%	40	%			%
beta-BHC	319-85-7	1.70	µg/Kg	1.10	µg/Kg	43	153	%	40	%	27	160	%	40	%			%
DCB Decachlorobiphe	2051-24-3		µg/Kg		µg/Kg			%		%			%		%	41	157	%
delta-BHC	319-86-8	1.70	µg/Kg	1.20	µg/Kg	54	152	%	40	%	10	160	%	40	%			%
Dieldrin	60-57-1	1.70	µg/Kg	0.470	µg/Kg	51	154	%	40	%	37	160	%	40	%			%
Endosulfan I	959-98-8	1.70	µg/Kg	0.520	µg/Kg	40	148	%	40	%	10	160	%	40	%			%
Endosulfan II	33213-65-9	1.70	µg/Kg	0.820	µg/Kg	42	137	%	40	%	16	150	%	40	%			%
Endosulfan sulfate	1031-07-8	1.70	µg/Kg	0.870	µg/Kg	50	153	%	40	%	10	160	%	40	%			%
Endrin	72-20-8	1.70	µg/Kg	0.500	µg/Kg	55	147	%	40	%	41	160	%	40	%			%
Endrin aldehyde	7421-93-4	1.70	µg/Kg	1.00	µg/Kg	43	158	%	40	%	10	160	%	38	%			%
Endrin ketone	53494-70-5	1.70	µg/Kg	0.630	µg/Kg	41	142	%	40	%	11	160	%	40	%			%

gamma-BHC (Lindane	58-89-9	1.70	µg/Kg	0.740	µg/Kg	44	160	%	40	%	18	160	%	40	%			%
gamma-Chlordane	5103-74-2	1.70	µg/Kg	0.420	µg/Kg	47	156	%	40	%	33	160	%	40	%			%
Heptachlor	76-44-8	1.70	µg/Kg	1.10	µg/Kg	47	137	%	40	%	26	160	%	40	%			%
Heptachlor epoxide	1024-57-3	1.70	µg/Kg	0.800	µg/Kg	53	153	%	40	%	43	160	%	29	%			%
Methoxychlor	72-43-5	3.30	µg/Kg	1.50	µg/Kg	40	152	%	40	%	10	160	%	39	%			%
Toxaphene	8001-35-2	67.0	µg/Kg	19.0	µg/Kg	40	160	%	40	%			%		%			%
Tetrachloro-m-xylene	877-09-8		µg/Kg		µg/Kg			%		%			%		%	40	149	%

Soils	Soxhlet Extraction	3540C
Soils	Herbicides (GC)	8151A

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	SREC - Recovery Lo	SREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	UREC - Recovery Lo	UREC - Recovery Hig	SUREC - Units
2,4-D	94-75-7	80.0	µg/Kg	19.0	µg/Kg	54	129	%	40	%	45	123	%	25	%			%
2,4,5-T	93-76-5	20.0	µg/Kg	3.75	µg/Kg	49	134	%	40	%	37	128	%	32	%			%
Silvex (2,4,5-TP)	93-72-1	20.0	µg/Kg	4.11	µg/Kg	53	120	%	40	%	41	120	%	27	%			%
2,4-Dichlorophenylace	19719-28-9		µg/Kg		µg/Kg			%		%			%		%	38	120	%

Soils	Extraction (Herbicides	8151A_SP
Soils	Dioxins and Furans (H	8290

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	SREC - Recovery Lo	SREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	UREC - Recovery Lo	UREC - Recovery Hig	SUREC - Units
2,3,7,8-TCDD	1746-01-6	1.00	pg/g			60	138	%	20	%	60	138	%	20	%			
2,3,7,8-TCDF	51207-31-9	1.00	pg/g			56	158	%	20	%	56	158	%	20	%			
1,2,3,7,8-PeCDD	40321-76-4	5.00	pg/g			70	122	%	20	%	70	122	%	20	%			
1,2,3,7,8-PeCDF	57117-41-6	5.00	pg/g			69	134	%	20	%	69	134	%	20	%			
2,3,4,7,8-PeCDF	57117-31-4	5.00	pg/g			70	131	%	20	%	70	131	%	20	%			
1,2,3,4,7,8-HxCDD	39227-28-6	5.00	pg/g			60	138	%	20	%	60	138	%	20	%			
1,2,3,6,7,8-HxCDD	57653-85-7	5.00	pg/g			68	136	%	20	%	68	136	%	20	%			
1,2,3,7,8,9-HxCDD	19408-74-3	5.00	pg/g			68	138	%	20	%	68	138	%	20	%			
1,2,3,4,7,8-HxCDF	70648-26-9	5.00	pg/g			74	128	%	20	%	74	128	%	20	%			
1,2,3,6,7,8-HxCDF	57117-44-9	5.00	pg/g			67	140	%	20	%	67	140	%	20	%			
2,3,4,6,7,8-HxCDF	60851-34-5	5.00	pg/g			71	137	%	20	%	71	137	%	20	%			
1,2,3,7,8,9-HxCDF	72918-21-9	5.00	pg/g			72	134	%	20	%	72	134	%	20	%			
1,2,3,4,6,7,8-HpCDD	35822-46-9	5.00	pg/g			71	128	%	20	%	71	128	%	20	%			
1,2,3,4,6,7,8-HpCDF	67562-39-4	5.00	pg/g			71	134	%	20	%	71	134	%	20	%			
1,2,3,4,7,8,9-HpCDF	55673-89-7	5.00	pg/g			68	129	%	20	%	68	129	%	20	%			
OCDD	3268-87-9	10.0	pg/g			70	128	%	20	%	70	128	%	20	%			
OCDF	39001-02-0	10.0	pg/g			63	141	%	20	%	63	141	%	20	%			
Total TCDD	41903-57-5	1.00	pg/g					%		%			%		%			
Total TCDF	30402-14-3	1.00	pg/g					%		%			%		%			
Total PeCDD	36088-22-9	5.00	pg/g					%		%			%		%			
Total PeCDF	30402-15-4	5.00	pg/g					%		%			%		%			
Total HxCDD	34465-46-8	5.00	pg/g					%		%			%		%			
Total HxCDF	55684-94-1	5.00	pg/g					%		%			%		%			
Total HpCDD	37871-00-4	5.00	pg/g					%		%			%		%			
Total HpCDF	38998-75-3	5.00	pg/g					%		%			%		%			
13C-2,3,7,8-TCDD	76523-40-5		pg/g			40	135	%		%			%		%			
13C-2,3,7,8-TCDF	89059-46-1		pg/g			40	135	%		%			%		%			
13C-1,2,3,7,8-PeCDD	109719-79-1		pg/g			40	135	%		%			%		%			
13C-1,2,3,7,8-PeCDF	109719-77-9		pg/g			40	135	%		%			%		%			
13C-1,2,3,6,7,8-HxCD	109719-81-5		pg/g			40	135	%		%			%		%			
13C-1,2,3,4,7,8-HxCD	114423-98-2		pg/g			40	135	%		%			%		%			
13C-1,2,3,4,6,7,8-HpC	109719-83-7		pg/g			40	135	%		%			%		%			
13C-1,2,3,4,6,7,8-HpC	109719-84-8		pg/g			40	135	%		%			%		%			
13C-OCDD	114423-97-1		pg/g			40	135	%		%			%		%			

Soils	Soxhlet Extraction of
TCLP Solids	Semivolatile Organic

8290_P_Sox	
8270C	

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	UREC - Recovery Lo	UREC - Recovery Hig	SUREC - Units
1,4-Dichlorobenzene	106-46-7	0.00400	mg/L	0.000340	mg/L	52	110	%	30	%	31	110	%	30	%			%
2,4,5-Trichlorophenol	95-95-4	0.0200	mg/L	0.000300	mg/L	51	110	%	30	%	41	110	%	30	%			%
2,4,6-Trichlorophenol	88-06-2	0.0200	mg/L	0.000240	mg/L	46	110	%	30	%	35	110	%	30	%			%
2,4-Dinitrotoluene	121-14-2	0.0200	mg/L	0.000250	mg/L	54	110	%	30	%	42	110	%	30	%			%
Hexachlorobenzene	118-74-1	0.0200	mg/L	0.0000852	mg/L	50	110	%	30	%	42	110	%	30	%			%
Hexachlorobutadiene	87-68-3	0.0200	mg/L	0.000270	mg/L	34	110	%	30	%	28	110	%	30	%			%
Hexachloroethane	67-72-1	0.0200	mg/L	0.000190	mg/L	41	110	%	30	%	26	110	%	30	%			%
3 & 4 Methylphenol	15831-10-4	0.0400	mg/L	0.000800	mg/L	48	110	%	30	%	29	110	%	30	%			%
2-Methylphenol	95-48-7	0.00400	mg/L	0.000170	mg/L	44	111	%	30	%	33	112	%	30	%			%
Nitrobenzene	98-95-3	0.00400	mg/L	0.0000400	mg/L	40	110	%	30	%	32	110	%	30	%			%
Pentachlorophenol	87-86-5	0.0400	mg/L	0.000270	mg/L	12	110	%	30	%	10	124	%	30	%			%
Pyridine	110-86-1	0.0200	mg/L	0.000350	mg/L	30	110	%	30	%	21	110	%	30	%			%
2-Fluorobiphenyl (Sur	321-60-8		mg/L		mg/L			%		%			%		%	30	110	%
2-Fluorophenol (Surr)	367-12-4		mg/L		mg/L			%		%			%		%	20	110	%
2,4,6-Tribromophenol	118-79-6		mg/L		mg/L			%		%			%		%	23	110	%
Nitrobenzene-d5 (Sur	4165-60-0		mg/L		mg/L			%		%			%		%	28	110	%
Phenol-d5 (Surr)	4165-62-2		mg/L		mg/L			%		%			%		%	21	110	%
Terphenyl-d14 (Surr)	1718-51-0		mg/L		mg/L			%		%			%		%	48	110	%

TCLP Solids	Liquid-Liquid Extractio
TCLP Solids	TCLP Extraction
TCLP Solids	Organochlorine Pestic

3510C	
1311_T	
8081A	

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	UREC - Recovery Lo	UREC - Recovery Hig	SUREC - Units
Chlordane (technical)	57-74-9	0.00500	mg/L	0.0000330	mg/L			%	35	%			%		%			%
Endrin	72-20-8	0.000500	mg/L	0.0000110	mg/L	73	146	%	35	%	47	140	%	50	%			%
Heptachlor	76-44-8	0.000500	mg/L	0.00000800	mg/L	60	140	%	35	%	44	129	%	50	%			%
Heptachlor epoxide	1024-57-3	0.000500	mg/L	0.00000710	mg/L	73	158	%	35	%	48	146	%	50	%			%
gamma-BHC (Lindane	58-89-9	0.000500	mg/L	0.00000640	mg/L	63	157	%	35	%	36	146	%	50	%			%
Methoxychlor	72-43-5	0.00100	mg/L	0.0000320	mg/L	49	160	%	35	%	35	152	%	50	%			%
Toxaphene	8001-35-2	0.0200	mg/L	0.000320	mg/L			%	35	%			%		%			%
Tetrachloro-m-xylene	877-09-8		mg/L		mg/L			%		%			%		%	40	129	%
DCB Decachlorobiphe	2051-24-3		mg/L		mg/L			%		%			%		%	40	152	%

TCLP Solids	Liquid-Liquid Extractio
TCLP Solids	TCLP Extraction
TCLP Solids	Herbicides (GC)

3510C	
1311_T	
8151A	

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	UREC - Recovery Lo	UREC - Recovery Hig	SUREC - Units
2,4-D	94-75-7	0.00400	mg/L	0.000410	mg/L	50	120	%	35	%	44	124	%	35	%			%
Silvex (2,4,5-TP)	93-72-1	0.00100	mg/L	0.000200	mg/L	45	129	%	35	%	35	135	%	35	%			%
2,4-Dichlorophenylace	19719-28-9		mg/L		mg/L			%		%			%		%	56	120	%

TCLP Solids	Extraction (Herbicides
TCLP Solids	TCLP Extraction
TCLP Solids	Metals (ICP)

8151A_AP	
1311_T	
6010B	

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	UREC - Recovery Lo	UREC - Recovery Hig	SUREC - Units
Arsenic	7440-38-2	0.500	mg/L	0.00320	mg/L	50	150	%	20	%	50	150	%	20	%			
Barium	7440-39-3	10.0	mg/L	0.000670	mg/L	50	150	%	20	%	50	150	%	20	%			
Cadmium	7440-43-9	0.100	mg/L	0.000660	mg/L	50	150	%	20	%	50	150	%	20	%			
Chromium	7440-47-3	0.500	mg/L	0.00220	mg/L	50	150	%	20	%	50	150	%	20	%			
Lead	7439-92-1	0.500	mg/L	0.00190	mg/L	50	150	%	20	%	50	150	%	20	%			
Selenium	7782-49-2	0.250	mg/L	0.00410	mg/L	50	150	%	20	%	50	150	%	20	%			
Silver	7440-22-4	0.500	mg/L	0.00220	mg/L	50	150	%	20	%	50	150	%	20	%			

TCLP Solids	Preparation, Total Me	3010A
TCLP Solids	TCLP Extraction	1311T_M
TCLP Solids	Mercury (CVAA)	7470A

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Mercury	7439-97-6	0.00200	mg/L	0.000120	mg/L	50	150	%	20	%	50	150	%	20	%			

TCLP Solids	Preparation, Mercury	7470A_Prep
TCLP Solids	TCLP Extraction	1311T_Hg
TCLP Solids	pH	9045C

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
pH	STL00204	0.100	SU			97	103	%	20	%								

TCLP Solids	Ignitability, Pensky-Ma	1010

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Flashpoint	STL00152	1.00	Degrees F			97	103	%										

TCLP Solids	Cyanide, Total and/or	9012A

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Cyanide, Total	57-12-5	0.500	mg/Kg	0.100	mg/Kg	68	123	%	20	%	50	134	%	20	%			

TCLP Solids	Cyanide, Total and/or	9012A_Prep
TCLP Solids	Sulfide, Acid soluble a	9034_Calc

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Sulfide	18496-25-8	30.0	mg/Kg	22.0	mg/Kg	70	130	%	20	%	10	154	%	20	%			

TCLP Solids	Sulfide, Distillation (Ac	9030B
TCLP Solids	Volatile Organic Comp	8260B

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
1,2-Dichloroethane-d4	17060-07-0		mg/L		mg/L			%	30	%			%	30	%	80	121	%
4-Bromofluorobenzen	460-00-4		mg/L		mg/L			%	30	%			%	30	%	70	124	%
Toluene-d8 (Surr)	2037-26-5		mg/L		mg/L			%	30	%			%	30	%	90	115	%
Dibromofluoromethan	1868-53-7		mg/L		mg/L			%	30	%			%	30	%	84	128	%
1,1-Dichloroethene	75-35-4	0.0250	mg/L	0.00950	mg/L	71	133	%	30	%	67	139	%	30	%			%
1,2-Dichloroethane	107-06-2	0.0250	mg/L	0.0110	mg/L	81	114	%	30	%	80	115	%	30	%			%
2-Butanone (MEK)	78-93-3	0.250	mg/L	0.0285	mg/L	49	120	%	30	%	49	117	%	30	%			%
Benzene	71-43-2	0.0250	mg/L	0.00650	mg/L	84	120	%	30	%	85	119	%	30	%			%
Carbon tetrachloride	56-23-5	0.0250	mg/L	0.00650	mg/L	54	122	%	30	%	60	110	%	30	%			%
Chlorobenzene	108-90-7	0.0250	mg/L	0.00750	mg/L	86	111	%	30	%	85	113	%	30	%			%
Chloroform	67-66-3	0.0250	mg/L	0.00800	mg/L	87	123	%	30	%	86	124	%	30	%			%
Tetrachloroethene	127-18-4	0.0250	mg/L	0.0145	mg/L	79	134	%	30	%	74	138	%	30	%			%
Trichloroethene	79-01-6	0.0250	mg/L	0.00850	mg/L	78	130	%	30	%	75	134	%	30	%			%
Vinyl chloride	75-01-4	0.0250	mg/L	0.0110	mg/L	56	111	%	30	%	51	118	%	30	%			%

TCLP Solids	Purge and Trap	5030B_Leach
TCLP Solids	TCLP Extraction	1311_Z
TCLP Waters	Volatile Organic Comp	8260B

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
1,2-Dichloroethane-d4	17060-07-0		mg/L		mg/L			%	30	%			%	30	%	80	121	%
4-Bromofluorobenzen	460-00-4		mg/L		mg/L			%	30	%			%	30	%	70	124	%
Toluene-d8 (Surr)	2037-26-5		mg/L		mg/L			%	30	%			%	30	%	90	115	%
Dibromofluoromethan	1868-53-7		mg/L		mg/L			%	30	%			%	30	%	84	128	%
1,1-Dichloroethene	75-35-4	0.0250	mg/L	0.00950	mg/L	71	133	%	30	%	67	139	%	30	%			%
1,2-Dichloroethane	107-06-2	0.0250	mg/L	0.0110	mg/L	81	114	%	30	%	80	115	%	30	%			%
2-Butanone (MEK)	78-93-3	0.250	mg/L	0.0285	mg/L	49	120	%	30	%	49	117	%	30	%			%
Benzene	71-43-2	0.0250	mg/L	0.00650	mg/L	84	120	%	30	%	85	119	%	30	%			%
Carbon tetrachloride	56-23-5	0.0250	mg/L	0.00650	mg/L	54	122	%	30	%	60	110	%	30	%			%
Chlorobenzene	108-90-7	0.0250	mg/L	0.00750	mg/L	86	111	%	30	%	85	113	%	30	%			%

Chloroform	67-66-3	0.0250	mg/L	0.00800	mg/L	87	123	%	30	%	86	124	%	30	%			%
Tetrachloroethene	127-18-4	0.0250	mg/L	0.0145	mg/L	79	134	%	30	%	74	138	%	30	%			%
Trichloroethene	79-01-6	0.0250	mg/L	0.00850	mg/L	78	130	%	30	%	75	134	%	30	%			%
Vinyl chloride	75-01-4	0.0250	mg/L	0.0110	mg/L	56	111	%	30	%	51	118	%	30	%			%

TCLP Waters	Purge and Trap	5030B_Leach
TCLP Waters	TCLP Extraction	1311_Z
TCLP Waters	Semivolatile Organic	8270C

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Lo	LCSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
1,4-Dichlorobenzene	106-46-7	0.00400	mg/L	0.000340	mg/L	52	110	%	30	%	31	110	%	30	%			%
2,4,5-Trichlorophenol	95-95-4	0.0200	mg/L	0.000300	mg/L	51	110	%	30	%	41	110	%	30	%			%
2,4,6-Trichlorophenol	88-06-2	0.0200	mg/L	0.000240	mg/L	46	110	%	30	%	35	110	%	30	%			%
2,4-Dinitrotoluene	121-14-2	0.0200	mg/L	0.000250	mg/L	54	110	%	30	%	42	110	%	30	%			%
Hexachlorobenzene	118-74-1	0.0200	mg/L	0.0000852	mg/L	50	110	%	30	%	42	110	%	30	%			%
Hexachlorobutadiene	87-68-3	0.0200	mg/L	0.000270	mg/L	34	110	%	30	%	28	110	%	30	%			%
Hexachloroethane	67-72-1	0.0200	mg/L	0.000190	mg/L	41	110	%	30	%	26	110	%	30	%			%
3 & 4 Methylphenol	15831-10-4	0.0400	mg/L	0.000800	mg/L	48	110	%	30	%	29	110	%	30	%			%
2-Methylphenol	95-48-7	0.00400	mg/L	0.000170	mg/L	44	111	%	30	%	33	112	%	30	%			%
Nitrobenzene	98-95-3	0.00400	mg/L	0.0000400	mg/L	40	110	%	30	%	32	110	%	30	%			%
Pentachlorophenol	87-86-5	0.0400	mg/L	0.000270	mg/L	12	110	%	30	%	10	124	%	30	%			%
Pyridine	110-86-1	0.0200	mg/L	0.000350	mg/L	30	110	%	30	%	21	110	%	30	%			%
2-Fluorobiphenyl (Sur	321-60-8		mg/L		mg/L			%		%			%		%	30	110	%
2-Fluorophenol (Surr)	367-12-4		mg/L		mg/L			%		%			%		%	20	110	%
2,4,6-Tribromophenol	118-79-6		mg/L		mg/L			%		%			%		%	23	110	%
Nitrobenzene-d5 (Sur	4165-60-0		mg/L		mg/L			%		%			%		%	28	110	%
Phenol-d5 (Surr)	4165-62-2		mg/L		mg/L			%		%			%		%	21	110	%
Terphenyl-d14 (Surr)	1718-51-0		mg/L		mg/L			%		%			%		%	48	110	%

TCLP Waters	Liquid-Liquid Extractio	3510C
TCLP Waters	TCLP Extraction	1311_T
TCLP Waters	Organochlorine Pestic	8081A

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Lo	LCSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Chlordane (technical)	57-74-9	0.00500	mg/L	0.0000330	mg/L			%	35	%			%		%			%
Endrin	72-20-8	0.000500	mg/L	0.0000110	mg/L	73	146	%	35	%	47	140	%	50	%			%
Heptachlor	76-44-8	0.000500	mg/L	0.00000800	mg/L	60	140	%	35	%	44	129	%	50	%			%
Heptachlor epoxide	1024-57-3	0.000500	mg/L	0.00000710	mg/L	73	158	%	35	%	48	146	%	50	%			%
gamma-BHC (Lindane	58-89-9	0.000500	mg/L	0.00000640	mg/L	63	157	%	35	%	36	146	%	50	%			%
Methoxychlor	72-43-5	0.00100	mg/L	0.0000320	mg/L	49	160	%	35	%	35	152	%	50	%			%
Toxaphene	8001-35-2	0.0200	mg/L	0.000320	mg/L			%	35	%			%		%			%
Tetrachloro-m-xylene	877-09-8		mg/L		mg/L			%		%			%		%	40	129	%
DCB Decachlorobiphe	2051-24-3		mg/L		mg/L			%		%			%		%	40	152	%

TCLP Waters	Liquid-Liquid Extractio	3520C
TCLP Waters	TCLP Extraction	1311_T
TCLP Waters	Herbicides (GC)	8151A

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Lo	LCSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
2,4-D	94-75-7	0.00400	mg/L	0.000410	mg/L	50	120	%	35	%	44	124	%	35	%			%
Silvex (2,4,5-TP)	93-72-1	0.00100	mg/L	0.000200	mg/L	45	129	%	35	%	35	135	%	35	%			%
2,4-Dichlorophenylace	19719-28-9		mg/L		mg/L			%		%			%		%	56	120	%

TCLP Waters	Extraction (Herbicides	8151A_AP
TCLP Waters	TCLP Extraction	1311_T
TCLP Waters	Metals (ICP)	6010B

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	LCSREC - Recovery Lo	LCSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	SUREC - Recovery Hig	SUREC - Units
Arsenic	7440-38-2	0.500	mg/L	0.00320	mg/L	50	150	%	20	%	50	150	%	20	%			
Barium	7440-39-3	10.0	mg/L	0.000670	mg/L	50	150	%	20	%	50	150	%	20	%			
Cadmium	7440-43-9	0.100	mg/L	0.000660	mg/L	50	150	%	20	%	50	150	%	20	%			
Chromium	7440-47-3	0.500	mg/L	0.00220	mg/L	50	150	%	20	%	50	150	%	20	%			

Lead	7439-92-1	0.500	mg/L	0.00190	mg/L	50	150	%	20	%	50	150	%	20	%			
Selenium	7782-49-2	0.250	mg/L	0.00410	mg/L	50	150	%	20	%	50	150	%	20	%			
Silver	7440-22-4	0.500	mg/L	0.00220	mg/L	50	150	%	20	%	50	150	%	20	%			

TCLP Waters	Preparation, Total Me	3010A
TCLP Waters	TCLP Extraction	1311T_M
TCLP Waters	Mercury (CVAA)	7470A

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	UREC - Recovery Hig	SUREC - Units
Mercury	7439-97-6	0.00200	mg/L	0.000120	mg/L	50	150	%	20	%	50	150	%	20	%			

TCLP Waters	Preparation, Mercury	7470A_Prep
TCLP Waters	TCLP Extraction	1311T_Hg
TCLP Waters	pH	9040C

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	UREC - Recovery Hig	SUREC - Units
pH	STL00204	0.100	SU			97	103	%	20	%								

TCLP Waters	Ignitability, Pensky-Ma	1010

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	UREC - Recovery Hig	SUREC - Units
Flashpoint	STL00152	1.00	Degrees F			97	103	%										

TCLP Waters	Cyanide, Total and/or	9012A

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	UREC - Recovery Hig	SUREC - Units
Cyanide, Total	57-12-5	0.0100	mg/L	0.00320	mg/L	69	118	%	20	%	42	140	%	20	%			

TCLP Waters	Cyanide, Total and/or	9012A_Prep
TCLP Waters	Sulfide, Acid soluble a	9034_Calc

Analyte Description	CAS Number	RL - Limit	RL - Units	MDL - Limit	MDL - Units	CSREC - Recovery Lo	CSREC - Recovery Hig	LCSREC - Units	LCSRPD - Precision	LCSRPD - Units	MSREC - Recovery Lo	MSREC - Recovery Hig	MSREC - Units	MSRPD - Precision	MSRPD - Units	SUREC - Recovery Lo	UREC - Recovery Hig	SUREC - Units
Sulfide	18496-25-8	3.00	mg/L	0.940	mg/L	70	130	%	20	%	27	124	%	20	%			

Appendix B

Laboratory Reference Data
TestAmerica - Knoxville

TestAmerica Knoxville
TO-15
South Dayton Dump

Analyte	CAS #	ppb(v/v)		ug/m3	
		RL	MDL	RL	MDL
1,1,1-Trichloroethane	71-55-6	0.20	0.030	1.1	0.16
1,1,2,2-Tetrachloroethane	79-34-5	0.20	0.061	1.4	0.42
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	0.20	0.031	1.5	0.24
1,1,2-Trichloroethane	79-00-5	0.20	0.054	1.1	0.29
1,1-Dichloroethane	75-34-3	0.20	0.026	0.81	0.11
1,1-Dichloroethene	75-35-4	0.20	0.034	0.79	0.13
1,2,4-Trichlorobenzene	120-82-1	1.0	0.098	7.4	0.73
1,2,4-Trimethylbenzene	95-63-6	0.20	0.063	0.98	0.31
1,2-Dibromoethane (EDB)	106-93-4	0.20	0.044	1.5	0.34
1,2-Dichloro-1,1,2,2-tetrafluoroethane	76-14-2	0.20	0.032	1.4	0.22
1,2-Dichlorobenzene	95-50-1	0.20	0.070	1.2	0.42
1,2-Dichloroethane	107-06-2	0.20	0.047	0.81	0.19
1,2-Dichloropropane	78-87-5	0.20	0.052	0.92	0.24
1,3,5-Trimethylbenzene	108-67-8	0.20	0.065	0.98	0.32
1,3-Butadiene	106-99-0	0.40	0.064	0.88	0.14
1,3-Dichlorobenzene	541-73-1	0.20	0.065	1.2	0.39
1,4-Dichlorobenzene	106-46-7	0.20	0.064	1.2	0.38
1,4-Dioxane	123-91-1	0.50	0.080	1.8	0.29
2,2,4-Trimethylpentane	540-84-1	0.50	0.039	2.3	0.18
2-Butanone (MEK)	78-93-3	1.0	0.20	2.9	0.59
2-Chlorotoluene	95-49-8	0.40	0.063	2.1	0.33
2-Hexanone	591-78-6	0.50	0.058	2.0	0.24
3-Chloropropene	107-05-1	0.20	0.048	0.63	0.15
4-Ethyltoluene	622-96-8	0.40	0.066	2.0	0.32
4-Isopropyltoluene	99-87-6	0.20	0.057	1.1	0.31
4-Methyl-2-pentanone (MIBK)	108-10-1	0.50	0.045	2.0	0.18
Acetone	67-64-1	5.0	1.4	12	3.3
Benzene	71-43-2	0.20	0.056	0.64	0.18
Benzyl chloride	100-44-7	0.40	0.078	2.1	0.40
Bromodichloromethane	75-27-4	0.20	0.044	1.3	0.29
Bromoform	75-25-2	0.20	0.048	2.1	0.50
Bromomethane	74-83-9	0.20	0.032	0.78	0.12
Butane	106-97-8	0.40	0.073	0.95	0.17
Butylbenzene	104-51-8	0.40	0.046	2.2	0.25
Carbon disulfide	75-15-0	0.50	0.031	1.6	0.097
Carbon tetrachloride	56-23-5	0.20	0.038	1.3	0.24
Chlorobenzene	108-90-7	0.20	0.049	0.92	0.23
Chlorodifluoromethane	75-45-6	0.20	0.037	0.71	0.13
Chloroethane	75-00-3	0.20	0.035	0.53	0.092
Chloroform	67-66-3	0.20	0.038	0.98	0.19
Chloromethane	74-87-3	0.50	0.16	1.0	0.33
cis-1,2-Dichloroethene	156-59-2	0.20	0.060	0.79	0.24
cis-1,3-Dichloropropene	10061-01-5	0.20	0.074	0.91	0.34
Cyclohexane	110-82-7	0.50	0.040	1.7	0.14

TestAmerica Knoxville
TO-15
South Dayton Dump

Analyte	CAS #	ppb(v/v)		ug/m3	
		RL	MDL	RL	MDL
Dibromochloromethane	124-48-1	0.20	0.042	1.7	0.36
Dichlorodifluoromethane	75-71-8	0.20	0.068	0.99	0.34
Ethylbenzene	100-41-4	0.20	0.068	0.87	0.30
Heptane	142-82-5	0.50	0.047	2.0	0.19
Hexachlorobutadiene	87-68-3	1.0	0.078	11	0.83
Hexane	110-54-3	0.50	0.032	1.8	0.11
Isopropyl alcohol	67-63-0	2.0	0.094	4.9	0.23
Isopropylbenzene	98-82-8	0.40	0.060	2.0	0.29
Methyl methacrylate	80-62-6	0.50	0.079	2.0	0.32
Methyl tert-butyl ether	1634-04-4	1.0	0.17	3.6	0.61
Methylene Chloride	75-09-2	0.50	0.13	1.7	0.45
m-Xylene & p-Xylene	179601-23-	0.20	0.12	0.87	0.52
Naphthalene	91-20-3	0.50	0.090	2.6	0.47
o-Xylene	95-47-6	0.20	0.061	0.87	0.26
Propylbenzene	103-65-1	0.40	0.056	2.0	0.28
sec-Butylbenzene	135-98-8	0.40	0.064	2.2	0.35
Styrene	100-42-5	0.20	0.058	0.85	0.25
tert-Butyl alcohol	75-65-0	2.0	0.038	6.1	0.12
tert-Butylbenzene	98-06-6	0.50	0.066	2.7	0.36
Tetrachloroethene	127-18-4	0.20	0.040	1.4	0.27
Tetrahydrofuran	109-99-9	1.0	0.063	2.9	0.19
Toluene	108-88-3	0.20	0.12	0.75	0.45
trans-1,2-Dichloroethene	156-60-5	0.20	0.050	0.79	0.20
trans-1,3-Dichloropropene	10061-02-6	0.20	0.048	0.91	0.22
Trichloroethene	79-01-6	0.20	0.036	1.1	0.19
Trichlorofluoromethane	75-69-4	0.20	0.024	1.1	0.13
Vinyl bromide	593-60-2	0.20	0.035	0.87	0.15
Vinyl chloride	75-01-4	0.20	0.071	0.51	0.18

Appendix C

Chain of Custody Form

CHAIN OF CUSTODY RECORD

COC NO.: PL-24567

14496 Sheldon Road, Suite #200, Plymouth, Michigan 48170
(734) 453-5123 Fax: (734) 453-5201

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CONESTOGA-ROVERS & ASSOCIATES

Project No:	*Phase/Task Code:*	*Laboratory Name:*	*Lab Location:*	*SSOW No:*
Project Name:		*Lab Contact:*	*Lab Quote No.:*	*Cooler No.:*
Project Location:				*Carrier:*
Chemistry Contact:				*Airbill No:*
Sampler(s):				*Date Shipped:*

Item	*Sample I.D. No.* (Containers for each sample may be combined on one line)	*Date* (mm/dd or dd/mm)	*Time* (hh:mm)	*SAMPLE TYPE* Grab	*SAMPLE TYPE* Composite	*MATRIX* Air / Oil / Waste	*MATRIX* Water	*MATRIX* Soil	*MATRIX* Sediment	*MATRIX* Other	*CONTAINER QUANTITY & PRESERVATION* Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	Thiosulfate	Methanol/Water (Soil VOC)	EnCores 3x5-g, 1x25-g	Other:	*ANALYSIS REQUESTED*												MS/MSD Request	*Comments Special Instructions/ Conditions Of Receipt*
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TAT Required: ☐ 1 Day ☐ 2 Days ☐ 3 Days ☐ 1 Week ☐ Standard ☐ Other:

Lab Deliverables (Select): ☐ Summary ☐ Expanded ☐ Other: ☐ See SSOW

RELINQUISHED BY	COMPANY	DATE	TIME	RECEIVED BY	COMPANY	DATE	TIME
1.				1.			
2.				2.			
3.				3.			

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Appendix D

Laboratory Sample Analysis Standard Operating Procedures

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

North Canton

SOP No. NC-MS-019, Rev. 2
Effective Date: 02/17/11

Title: DETERMINATION OF VOLATILE ORGANICS BY GC/MS BASED ON METHODS 8260C, 8260B, AND 8260A

[Method: EPA Methods 8260C, 8260B, and 8260A]

Approvals (Signature/Date):

Technology Specialist	02/14/11 Date	Health & Safety Coordinator	02/01/11 Date
Quality Assurance Manager	02/01/11 Date	Technical Director	02/04/11 Date
Laboratory Director	02/09/11 Date		

This SOP was previously identified as SOP No. NC-MS-019, Rev 1, dated 01/07/09

Facility Distribution No. _____ **Distributed To: _____**

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1.0 SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Volatile Organic Compounds in waters, wastewater, soils, sludges, and other solid matrices.

1.2 This SOP is applicable to Methods 8260B and 8260C. It may also be used for analysis following Method 8260A.

1.3 This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4. The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 5 to 200 µg/L for 5 mL waters, 1 to 40 µg/L for low-level waters, 5 to 200 µg/kg for low-level soils, and 250 to 10,000 µg/kg for medium-level soils. Reporting limits are listed in Tables 1 and 3.

1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates, and laboratory control spike samples.

2.0 SUMMARY OF METHOD

2.1. Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2. Aqueous samples are purged directly. Soils are preserved by extracting the volatile analytes into methanol. Soil samples may also be preserved with sodium bisulfate or by freezing and purging directly.

2.3. In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature or at 40°C (40°C required for low-level soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbant column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components, which are detected with a mass spectrometer.

2.4. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified

component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3.0 DEFINITIONS

3.1 Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. All glassware is cleaned per SOP NC-QA-014. The use of ultra high purity gases, prepurged purified reagent water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants. In extreme cases, the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination. Refer to SOP NC-QA-020 for additional information on holding blanks.

4.3. Matrix interferences may be caused by non-target contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially on an autosampler. Whenever an unusually concentrated sample is analyzed, it must be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5. Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered, the sample must be diluted.

5. SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately. Cut-resistant gloves MUST be worn when opening VOA vials and when doing any other task that presents a strong possibility of getting cut.

5.3. Primary Materials Used

5.3.1. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sodium bisulfate	Irritant	None	Causes mild to severe irritation to the eyes. Prolonged exposure may cause burn if not flushed with water. May cause mild irritation to skin. Prolonged exposure may cause burn if not flushed with water.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. It is recommended that analysts break up work tasks to avoid repetitive motion tasks, such as opening a large number of vials or containers in one time period.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with a sticker that reads "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made. MS VOA samples may be prepared outside of the hood, unless it is known that concentrations are high.

5.6. The preparation of standards and reagents must be conducted in a fume hood with the sash closed as far as the operations will permit. MS VOA standards may be prepared outside of the hood due to low concentrations of analytes.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the laboratory Group Leader.

5.8. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the TestAmerica Corporate Environmental Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed by annual refresher training.

5.9. Specific Safety Concerns or Requirements

5.9.1. The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.9.2. The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.9.3. There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.9.4. Sodium bisulfate creates Sulfuric Acid when mixed with water.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes: 10 µL and larger

6.2. Syringe: 5, 25, or 50 mL glass with luer loktip, if applicable to the purging device.

6.3. Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.01 g

6.4. Glassware

6.4.1. Vials: 20 and 40 mL with screw caps and Teflon® liners.

6.4.2. Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.

6.5. Spatula: Stainless steel.

6.6. Disposable pipettes: Pasteur, 5 ¾ in.

6.7. pH paper: Wide range, pH 0-14.

6.8. Gases

6.8.1. Helium: Ultra high purity, gr. 5, 99.999%.

6.8.2. Nitrogen: Ultra high purity from cylinders or gas generators may be used as an alternative to helium for purge gas.

6.9. Purge and Trap Device. The purge and trap device consists of the sample purger, trap, and desorber.

6.9.1. Sample Purger. The recommended purging chamber is designed to accept 5 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low-level soils are purged directly from a VOA vial.

6.9.2. Trap. A variety of traps may be used, depending on the target analytes required. One of the traps used is the Vocarb 3000 trap. Other traps such as the OI 10 may be used if the Quality Control criteria are met. Refer also to instrument operating manuals located within the laboratory.

6.9.3. Desorber. The desorber must be capable of rapidly heating the trap to at least 180°C. Many such devices are commercially available.

6.9.4. Sample Heater. A heater capable of maintaining the purge device at 40°C is necessary for low-level soil analysis.

6.10. Gas Chromatograph/Mass Spectrometer System

6.10.1. Gas Chromatograph. The gas chromatograph (GC) system must be capable of temperature programming.

6.10.2. Gas Chromatographic Columns. Capillary columns are used. Some typical columns are listed below:

6.10.2.1. Column 1. 20m x 0.18 ID DB-624 with 1 µm film thickness.

6.10.2.2. Mass Spectrometer. The mass spectrometer must be capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-Bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.10.3. GC/MS Interface. In general, direct introduction to the mass spectrometer is used but any interface that achieves all acceptance criteria may be used.

6.10.4. Data System. A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The NIST/EPA mass spectral library must be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Methanol. Purge and Trap grade, high purity

7.1.2. Reagent Water. High purity water that meets the requirements for a method blank when analyzed (see Section 9.4). Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.

7.1.3. Hydrochloric Acid – (1:1 v/v). Reagent grade or equivalent

7.1.4. Sodium bisulfate. Reagent grade or equivalent

7.2. Standards

7.2.1. Calibration Standard

7.2.1.1. Stock Solutions. Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon®-sealed screw-cap bottles with minimal headspace at -10° to -20°C. Note that standard/spiking concentrations or vendors are subject to change.

7.2.1.2. Working standards. A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20% then corrective action is necessary. This may includes steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem, then a new initial calibration must be performed.

7.2.1.3. Aqueous Calibration Standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.2.1.4. If stock or secondary dilution standards are purchased in sealed ampoules, they may be used up to the manufacturer's expiration date.

7.2.1.5. Additional information can be found in SOP NC-QA-017.

7.2.2. Internal Standards. Internal standards are added to all samples, standards, and blank analyses. Refer to Table 5 for internal standard components.

7.2.3. Surrogate Standards. Refer to Table 6 for surrogate standard components and spiking levels.

7.2.4. Laboratory Control Sample Spiking Solutions. Refer to Table 7 for LCS components and spiking levels.

7.2.5. Matrix Spiking Solutions. The matrix spike contains the same components as the LCS. Refer to Table 7.

7.2.6. Tuning Standard. A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 50 ng/µL of 4-Bromofluorobenzene in methanol is prepared as described in Sections 7.2.1.1 and 7.2.1.2.

7.2.7. All standard preparation information is detailed in the Standard Logbook.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection to analysis.

8.2. For DoD samples, water samples are normally preserved at pH $\leq$ 2 with 1:1 hydrochloric acid. Unpreserved water samples must be analyzed within seven days of sampling.

8.3. Solid samples are field preserved with sodium bisulfate solution or by freezing upon receipt at the laboratory for low-level analysis, or with methanol for medium-level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Analysis must be completed 14 days from sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore™ sample. (The 5g or 25g sampler can be used, depending on client preference). Following shipment back to the lab, the soil is preserved in methanol. This is the medium level procedure. If very low detection limits are needed (< 50 µg/kg for most analytes), then it will be necessary to use two additional 5g EnCore™ samplers or to use field preservation.

8.5. Sample collection for medium level analysis using EnCore™ samplers

8.5.1. Ship one 5g (or 25g) EnCore™ sampler per field sample position.

8.5.2. An additional 2 oz plastic bottle must be shipped for percent moisture determination.

8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.5.4. Add the correct amount of surrogate spiking mixture. (Add 25 µL of 2500 µg/mL solution for a nominal 25g sample, 5 µL for a nominal 5g sample.) Refer to Section 17.2 for Michigan project criteria.

8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 500 µL of 50 µg/mL solution for a nominal 25 g sample, 100 µL for a nominal 5g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25g sample or 5 mL methanol for a nominal 5g sample. Refer to Section 17.2 for Michigan project criteria.

8.5.6. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (500 µL of spike to 25 mL methanol or 100 µL spike to 5 mL methanol). Refer to Section 17.2 for Michigan project criteria.

8.5.7. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.6. Sample collection for medium-level analysis using field methanol preservation

8.6.1. Prepare a 2-oz sample container by adding 25 mL purge and trap grade methanol. (If a 5g sample is to be used, add 5 mL methanol to a 2 oz container or VOA vial).

8.6.2. Seal the bottle and attach a label.

8.6.3. Weigh the bottle to the nearest 0.01 g, and note the weight on the label.

8.6.4. Ship with appropriate sampling instructions.

8.6.5. Each sample will require an additional 2 oz plastic bottle with no preservative for percent moisture determination.

8.6.6. At client request, the methanol addition and weighing may also be performed in the field.

8.6.7. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.6.8. Add the correct amount of surrogate spiking mixture. (Add 25 µL of 2500 µg/mL solution for a nominal 25g sample, 5 µL for a nominal 5g sample.) Refer to Section 17.2 for Michigan project criteria.

8.6.9. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 500 µL of 50 µg/mL solution for a nominal 25g sample, 100 µL for a nominal 5g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25g sample or 5 mL methanol for a nominal 5g sample. Refer to Section 17.2 for Michigan project criteria.

8.6.10. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (500 µL of spike to 25 mL methanol or 100 µL spike to 5 mL methanol). Refer to Section 17.2 for Michigan project criteria.

8.6.11. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.7. Low-level procedure

8.7.1. If low detection limits are required (typically < 50 µg/kg), low-level soil preservation must be used. However, it is also necessary to take a sample for the medium-level (field methanol preserved or using the EnCore™ sampler) procedure in case the concentration of analytes in the soil is above the calibration range of the low-level procedure.

8.7.2. A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method. (Varian Archon or O.I. 4552).

8.7.3. The soil sample is taken using a 5g EnCore™ sampling device and returned to the lab. It is recommended that two EnCore™ samplers be used for each field sample position to allow for any reruns than may be necessary. A separate sample for % moisture determination is also necessary.

8.7.4. Prepare VOA vials for sodium bisulfate preservation by adding a magnetic stir bar, approximately 1 g of sodium bisulfate, and 5 mL of reagent water. Prepare vials for preservation by freezing by adding a stir bar and 5 mL reagent water.

8.7.5. Seal and label the vial. It is strongly recommended that the vial is labeled with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction. The label absolutely must not cover the neck of the vial or the autosampler will malfunction.

8.7.6. Weigh the vial to the nearest 0.01 g, and note the weight on the label.

8.7.7. Extrude the soil sample from the EnCore™ sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil, and note on the label.

Note: Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, add 5 mL of water instead, and freeze at <-10°C within 48 hours. The sample must be analyzed within 14 days after sampling and stored at a 45 degree angle in the freezer.

8.7.8. Alternatively the sodium bisulfate preservation may be performed in the field. This is not recommended because of the many problems that can occur in the field setting.

Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional 2 oz plastic bottle with no preservative for percent moisture determination, and an additional VOA vial preserved with methanol for the medium level procedure. Depending on the type of soil, it may also be necessary to ship vials with no or extra preservative.

8.8. Unpreserved Soils

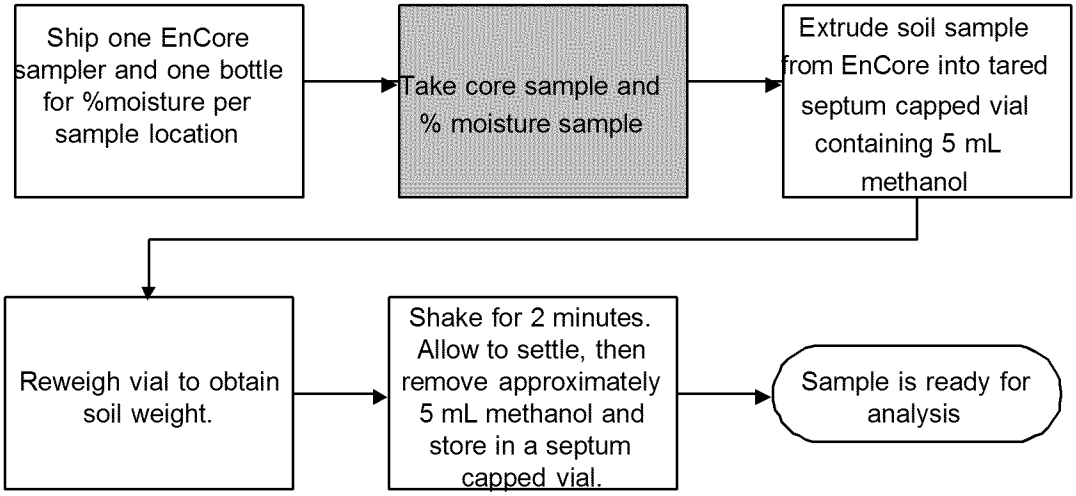
8.8.1. At specific client request, unpreserved soils packed into glass jars or brass tubes may be accepted and sub-sampled in the lab. This is the old procedure based on Method 5030A and Method 8260A. It is no longer included in SW846 and is likely to generate results that are biased low, possibly by more than an order of magnitude.

8.9. Aqueous samples are stored in glass containers with Teflon®-lined septa at 4°C ± 2°C with minimum headspace.

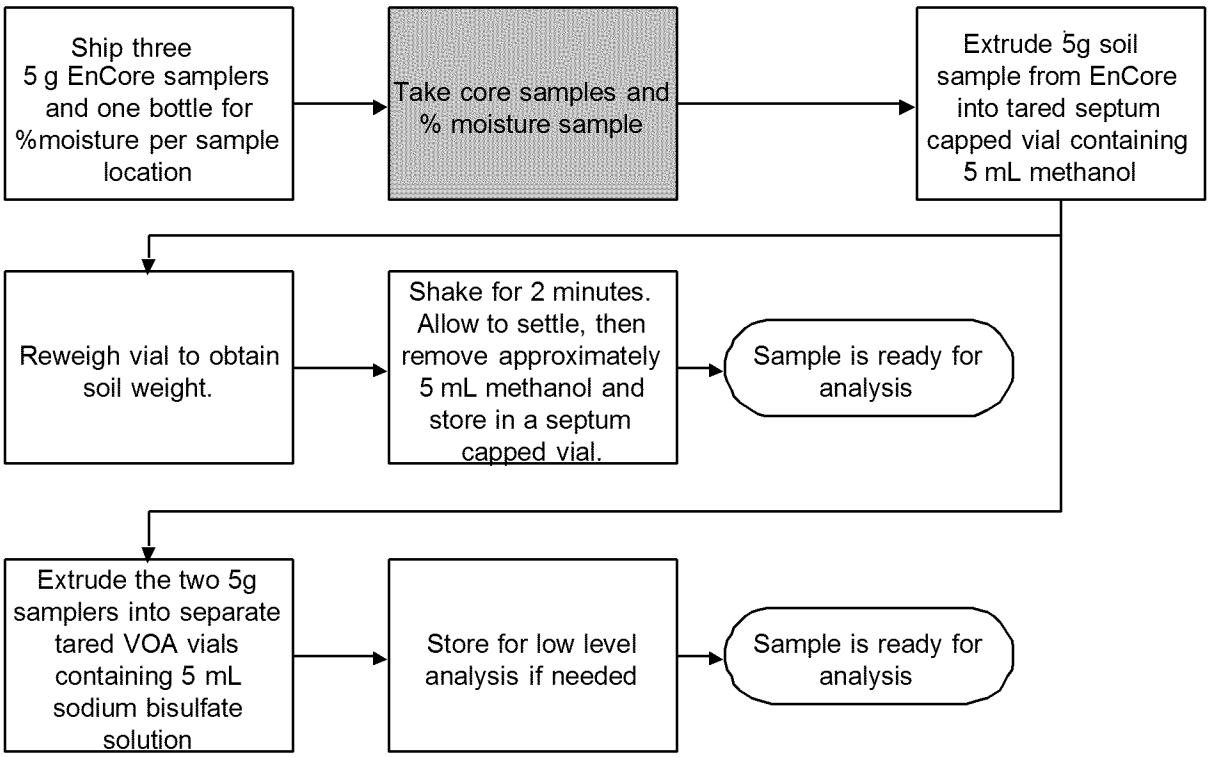
8.10. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14-day holding time. However, they should be analyzed as soon as possible. The lack of preservation must be addressed in the case narrative). Maximum holding time for the EnCore™ sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.

8.11. A holding blank is stored with the samples. This is analyzed weekly. It is replaced every seven days.

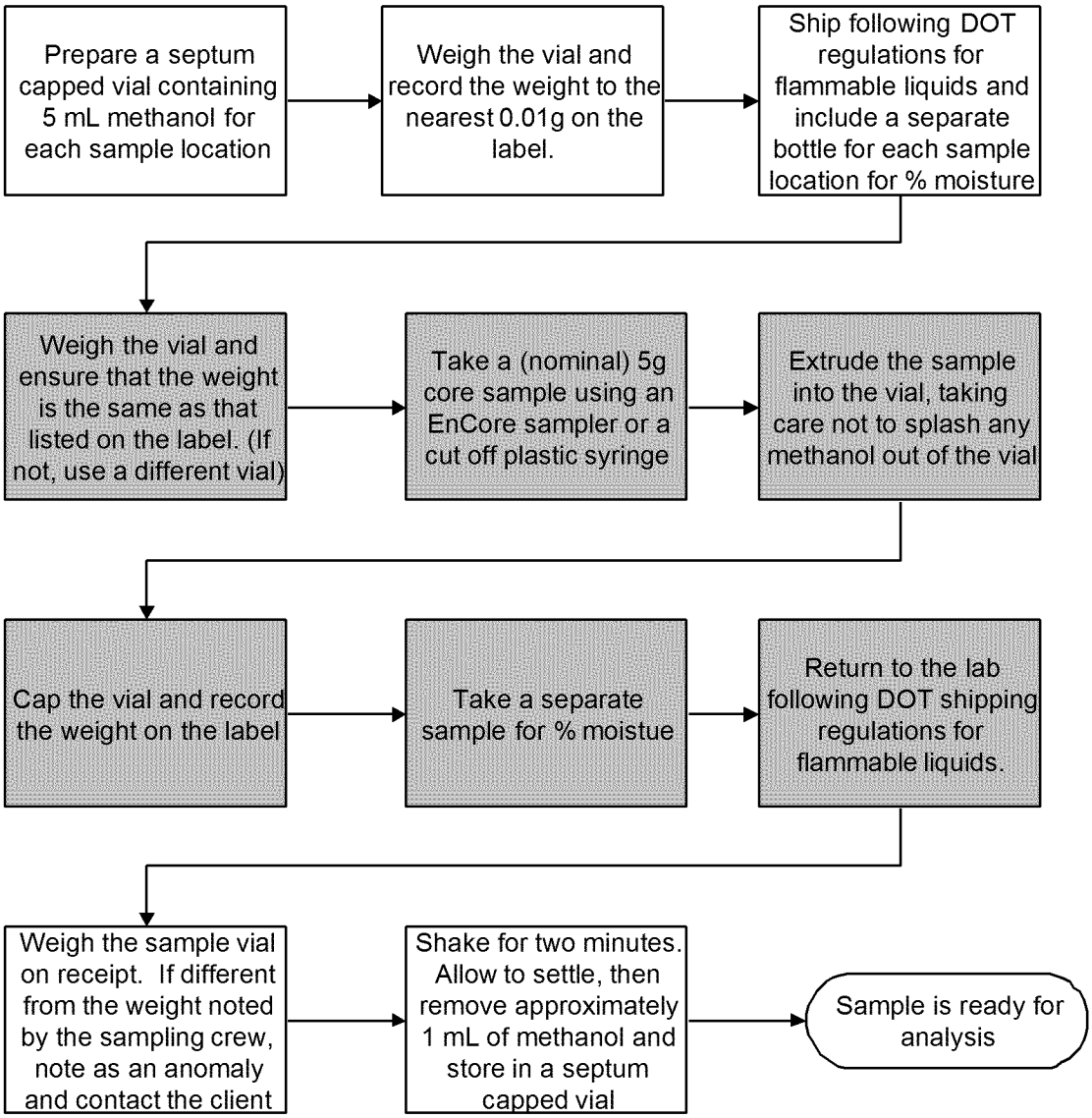
EnCore procedure when low level is not required (field steps in gray)



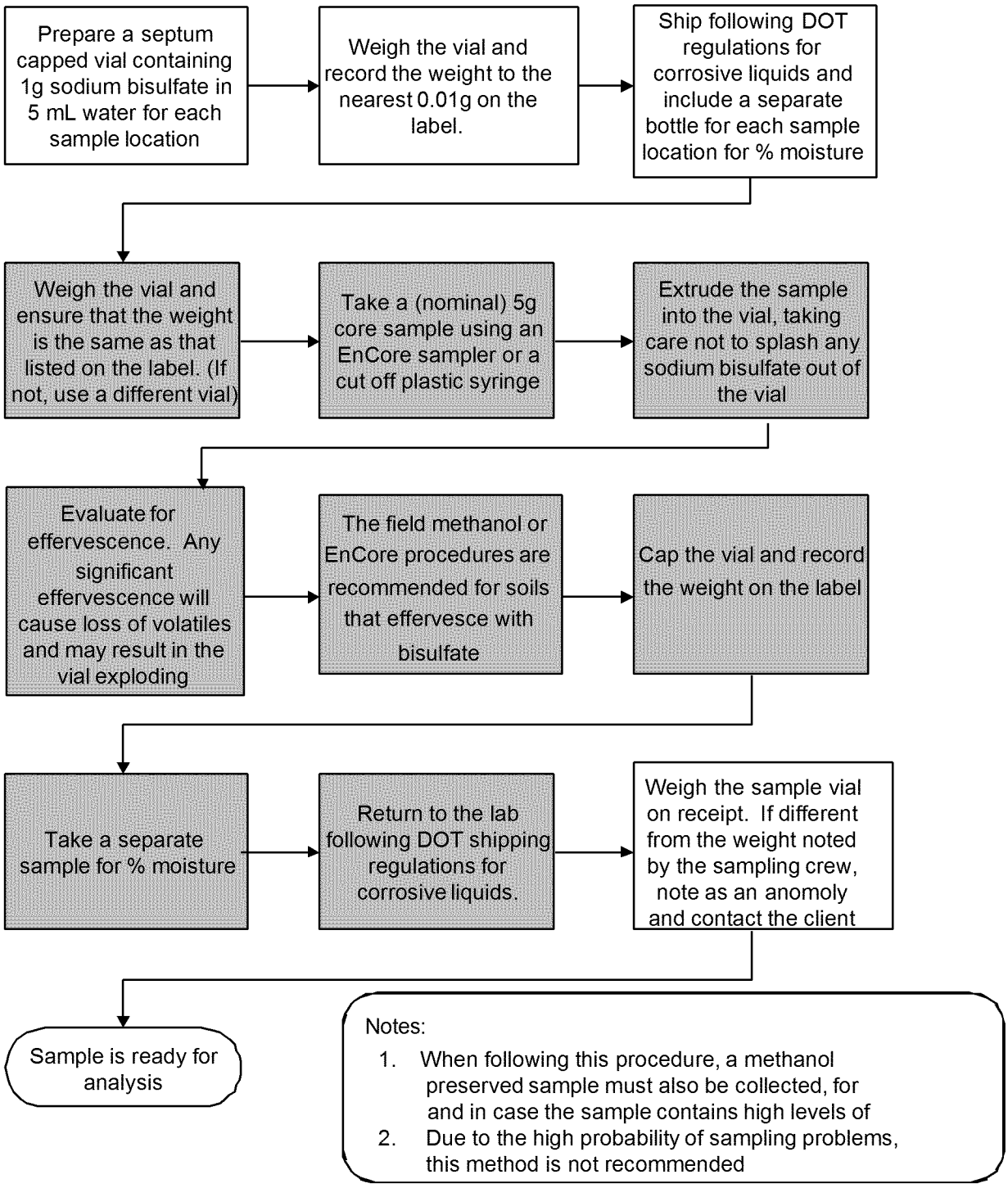
EnCore procedure when low level is required



Field methanol extraction procedure (field steps in gray)



Field bisulfate preservation procedure (field steps in gray)



9. QUALITY CONTROL

9.1. Batch

9.1.1. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this method, each BFB analysis will start a new batch. Batches for medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort must be made to keep the samples together.

9.1.1.1. The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Refer to the TestAmerica QC program document (QA-003) for further details of the batch definition.

9.2. Control Limits

9.2.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.2.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMs (QC Browser program).

9.3. Surrogates

9.3.1. Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Table 6. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure instrument performance is acceptable.
- Recalculate the data and/or re-analyze if either of the above checks reveal a problem.
- Reprepare and re-analyze the sample if there is sufficient volume. If there is insufficient volume, the surrogate is narrated.

It is only necessary to reprepare/re-analyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out-of-control results are not due to matrix effect.

9.3.2 If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and reprep aration is not necessary. If the sample is out of control and the MS and/or MSD is in control, then re-analysis or flagging of the data is required. For Ohio VAP samples, all surrogates must be in control, or samples must be reprepared and re-analyzed.

Note: For Ohio VAP and DoD samples, all surrogates must be within acceptance criteria. The exceptions for Ohio VAP are as follows: (a) insufficient sample for re-extraction, or (b) the surrogates are biased high and the samples are non-detect.

9.3.3 For concrete matrix, Dibromofluoromethane may have poor recovery in samples and matrix spikes. If the surrogate does not meet criteria, no further action is required due to matrix.

9.3.4 Refer to the TestAmerica QC Program document (QA-003) for further details of the corrective actions.

9.4 Method Blanks

9.4.1 For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. For low-level volatiles, the method blank consists of reagent water. For medium-level volatiles, the method blank consists of the same volume of methanol that was used to prepare the samples. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below). The method blank is acceptable if any compound detected in the blank is present in the associated samples at ten times the blank level. For Ohio VAP work, there can be no target analyte greater than the RL in the method blank unless the sample result is ND. All samples associated with an unacceptable blank will be reprepared and re-analyzed.

- If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone), the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

- Re-analysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers.

9.4.2 The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client must take place. For Ohio VAP samples, all surrogates must be in control, or repreparation of the batch is required.

9.4.3 If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B," and appropriate comments may be made in a narrative to provide further documentation.

9.4.4 Refer to the TestAmerica QC Program document, Policy QA-003, for further details of the corrective actions.

9.4.5 Refer to SOP NC-QA-016 for further details concerning DoD Project Work.

9.5 Laboratory Control Samples (LCS)

9.5.1 For each batch of samples, analyze an LCS. The LCS is analyzed after the calibration standard, and normally before any samples. The LCS contains a representative subset of the analytes of interest (see Table 7), and must contain the same analytes as the matrix spike. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be reprepareation and re-analysis of the batch. For Ohio VAP samples, all surrogates must be in control on the LCS, or reprepareation and re-analysis of the batch is required. The exceptions are as follows: (a) insufficient sample for reprepareation, (b) expired holding times, or (c) the LCS is biased high and the samples are non-detect for those analytes.

- If the batch is not re-extracted and re-analyzed, the reasons for accepting the batch must be clearly presented in the project records and the report.

- If re-extraction and re-analysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5.2 Refer to the TestAmerica QC Program document (Policy QA-003) for further details of the corrective action.

9.5.3 If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be

expected statistically. These requirements must be negotiated with the client. n-Hexane must be spiked and reported for the LCS for Ohio VAP samples.

9.5.4 If full analyte spike lists are used at the client request, it is possible some compounds in the LCS may interfere with each other. In that case, the lab will quantitate those compounds in the LCS with a secondary ion which is free from interferences.

9.6 Matrix Spikes

9.6.1 For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 7. Compare the percent recovery and relative percent difference (RPD) to that in the laboratory-specific, historically-generated limits.

9.6.2 If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

9.6.2.1 If the recovery for any component is outside QC limits for both the matrix spike/spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include re-analysis of the batch.

9.6.2.2 If an MS/MSD is not possible due to limited sample, then an LCS duplicate may be analyzed, if required by specific clients or program.

9.6.2.3 The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7 Nonconformance and Corrective Action

9.7.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked through the analysis of the 4-Bromofluoro-

benzene (BFB) to establish that a given GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations (analyzed under the same BFB tune), to determine the linearity of the response utilizing target calibration standards. Once the system has been calibrated, the calibration must be verified each twelve hour time period for each GC/MS system.

10.1.2. General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 AMU
Scan Time:	To give at least 5 scans/peak, but not to exceed 2 seconds/scan
Injector Temperature:	200–250°C
Source Temperature:	According to manufacturer's specifications
Transfer Line	Temperature: 250–300°C
Purge Flow:	40 mL/minute
Carrier Gas	Flow: 0.4 – 0.6 mL/minute

10.2 Gas chromatograph suggested temperature program

10.2.1 BFB Analysis

Initial Temperature:	100°C
Initial Hold Time:	0.1 minute
Temperature Program:	20°C/minute
Final Temperature:	200°C

10.2.2 Sample Analysis

Initial Temperature:	40°C
Initial Hold Time:	2 minutes
Temperature Program:	15°C/minute
Final Temperature:	200°C
Final Hold Time:	3 minutes

10.3. Instrument Tuning

10.3.1. Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 8 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each 12-hour time period. The 12-hour time period begins at the moment of injection of BFB.

10.4. Initial Calibration

10.4.1. A series of at least five initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Six standards must be used for a quadratic least squares calibration. Suggested calibration levels for a 5 mL purge are: 5, 20, 50, 100, and 200 µg/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Suggested calibration levels for a low level 5mL purge are 1, 5, 10, 20, and 40 µg/L. Again, some analytes are prepared at higher levels. Tables 2, 2A, and 4 list the calibration levels for each analyte. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument. (For example, adequate sensitivity can be obtained by using a 5 mL purge volume to reach the same reporting limits that once required a 25 mL purge. The calibration levels will still be the same 1, 5, 10, 20, 40 µg/L.) However, the same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

NOTE: For Method 8260C. Historically the surrogate compounds have been included in the multi-point initial calibration at variable concentrations in order to evaluate the linear response as with any target analyte. However, with improvements in instrumentation and more reliance on the autosampler, an option is available depending on the project-specific data quality requirements for allowing the autosampler (or using a manual technique) to spike the initial calibration standards with surrogates in the same manner as the samples are spiked. With this option the surrogate standards in the initial calibration can be averaged to develop a response factor and an effective one point calibration with the sole purpose to measure the surrogate recovery using the same concentration for each sample analysis. For this calibration option the surrogate linear response is less important, since multiple concentrations of surrogates are not being measured. Instead, the surrogate concentration remains constant throughout and the recovery of this known concentration can easily be attained without demonstrating if the response is linear. Under a second calibration option, the surrogates can be calibrated in the same manner as the target analytes, however, the laboratory should have the latitude to employ either option given the instrument system limitations and the ability to meet the project's data quality objectives.

10.4.2. It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for same tests.

10.4.3. Internal standard calibration is used. The internal standards are listed in Table 5. Target compounds must reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area

response of the characteristic ions against the concentration for each compound and internal standard. See Table 12 for a list of characteristic ions. See Equation 1, Section 12, for calculation of response factor.

10.4.4. For Method 8260B, the % RSD of the calibration check compounds (CCC) must be less than 30%. Refer to Table 11 for the CCCs. This criteria must be met before sample analysis begins.

10.4.4.1. Calibration Check Compound (CCC) (Method 8260B only)

10.4.4.1.1. CCCs are a representative group of compounds, which are used to evaluate initial calibrations and continuing calibrations Relative percent difference for the initial calibration and % drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

10.4.4.2. System Performance Check Compounds (SPCC) (Method 8260B only)

10.4.4.2.1 SPCCs are compounds, which are sensitive to system performance problems and are used to evaluate system performance and sensitivity A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

10.4.5. The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 9 for the SPCC compounds for Method 8260B and required minimum response factors. Refer to Table 10 for the recommended minimum relative response factor criteria for initial and continuing calibration verification for Method 8260C.

10.4.6. Weighting of Data Points

10.4.6.1. In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called 1/X^2 weighting) will improve accuracy at the low end of the curve and must be used if the data system has this capability. The Y-intercept is evaluated to determine calibration acceptability.

10.4.7. For any analyte with % RSD >15%, linear or quadratic curve fits may be used if the compounds have historically exhibited a non-linear response. The analyst must consider instrument maintenance to improve the linearity of response. Nonlinear calibration models cannot be used to extend the calibration range for compounds that normally exhibit a linear response, but in a narrower calibration range. If the % RSD is > 15%, the analyst may drop the low or high in the ICAL, as long as a minimum of five points are maintained (six points for quadratic) and the quantitation range is adjusted accordingly. Otherwise, the coefficient of determination r^2 must be $\geq$ 0.990. For Method 8260C, % RSD is ± 20% for each target analyte.

10.4.8. If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.9. The calibration standards for the initial five-point calibration for low-level soils that are not preserved in sodium bisulfate (i.e., are preserved by freezing or not preserved) must be heated to 40° C for purging. Using this calibration curve for water samples is acceptable as long as all calibration, QC, and samples are also heated to 40° C. A separate five-point calibration must be prepared for analysis of low level soils that are preserved with sodium bisulfate. Low-level soils analysis requires the use of a closed vial autosampler such as the Varian Archon, O.I. 4552 or Tekmar Precept. Each standard for analysis of sodium bisulfate preserved samples is prepared by spiking the methanolic standard solution through the septum of a VOA vial containing 5 mL of water and 1g sodium bisulfate. The standards are heated to 40° C for purging. All low-level soil samples, standards, and blanks must also be heated to 40° C for purging.

10.4.10. Non-standard analytes are sometimes requested. For these analytes, it is acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five-point initial calibration. If the analyte is detected in any of the samples, a five-point initial calibration must be generated and the sample(s) re-analyzed for quantitation. However, if the analyte is not detected, the non-detect must be reported and no further action is necessary.

Note: This procedure must not be used for Ohio VAP samples.

10.4.11. Calibration accuracy is verified by analyzing a second source standard (ICV) immediately after the initial calibration. For Method 8260B, the recovery for CCC compounds must be ≤ 20%. The recovery for non-CCC compounds must be ≤ 50% with an allowance of up to six compounds > 50%.

10.4.11.1 For Method 8260C, the acceptance criteria is 70-130% for each target analyte.

10.5. Continuing Calibration. The initial calibration must be verified every 12 hours.

10.5.1. Continuing calibration begins with analysis of BFB as described in Section 10.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. A midpoint calibration standard is used as the continuing calibration.

10.5.2. The RF data from the standards are compared with the average RF from the initial five-point calibration to determine the percent drift of the CCC compounds. The calculation is given in Equation 4, Section 12.3.4.

10.5.3. For Method 8260B, the % drift of the CCCs must be ≤ 20% for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 9. In addition, the percent drift of all analytes must be ≤ 50% with allowance for up to six target analytes to have percent drift > 50%.

10.5.3.1. For Method 8260C, all compounds of interest must be verified at 20%.

10.5.3.2. Refer to Table 11 for specific Ohio VAP analytes.

10.5.4. If the CCCs and/or the SPCCs do not meet the criteria in Section 10.5.3 and Table 9, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective actions such as a different type of column will require a new initial calibration. For Method 8260C, any sample non-detects for an analyte that fails the SOP criteria low, must have a low level CCV (CCV at the RL) in the batch as a sensitivity demonstration. The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detect samples to be reported without flagging.

10.5.5. Once the above criteria have been met, sample analysis may begin. **Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs.** Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample *desorbed* less than or equal to 12 hours after the BFB is acceptable.)

11. PROCEDURE

11.1. Procedural Variations

11.1.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation must be completely documented using a Nonconformance Memo and approved by a Supervisor or Group

Leader and QA Manager. The Nonconformance Memo must be filed in the project file.

11.1.2. Any unauthorized deviations from this procedure must also be documented as a non-conformance with a cause and corrective action described. The laboratory may not deviate from the method for Ohio VAP samples.

11.2. Preliminary Evaluation

11.2.1. Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories.

11.3. Sample Analysis Procedure

11.3.1. All analysis conditions for samples must be the same as for the continuing calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.3.2. All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain an MS/MSD, an LCS, and a method blank. See Section 9.4 for method blank preparation.

11.3.2.1. If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next tune period. However, if any re-tuning of the instrument is necessary, or if a period of greater than 24 hours from the preceding BFB tune has passed, a new batch must be started. For medium-level soils, the batch is defined at the sample preparation stage.

11.3.2.2. It is not necessary to re-analyze batch QC with re-analyses of samples. However, any reruns must be part of a valid batch.

11.3.3 Dilutions must be done just prior to the GC/MS analysis of the sample. Dilutions are made in a Luerlok syringe. Calculate the volume of reagent water required for the dilution. Fill the syringe with reagent water, compress the water to vent any residual air and adjust the water volume to the desired amount. Adjust the plunger to the mark and inject the proper aliquot of sample into the syringe. If the dilution required would use less than 1 µL of sample, then serial dilutions must be made in volumetric flasks. Dilutions may also be prepared in a 40 mL vial. An appropriate amount of water is added to the vial. The sample is added using an appropriate syringe.

11.3.3.1 The diluted concentration is to be estimated to be in the upper half of the calibration range.

11.4. Methanol Extract Soils

11.4.1 Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Sections 8.5 or 8.6) to the syringe. If less than 1 µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 1 µL will be added to the water in the syringe. Refer to Section 17.2 for Michigan project requirements.

11.5. Liquid wastes that are soluble in methanol and insoluble in water.

11.5.1 Pipette 1 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1 g.

11.5.2 Quickly add 4 mL of methanol, then add 5µL of a 2500 µg/mL surrogate spiking solution to bring the final volume to 5 mL. Cap the vial and shake for two minutes to mix thoroughly. For an MS/MSD or LCS, 4.9 mL of methanol, 5µL of a 2500 µg/mL surrogate spiking solution, and 0.1 mL of matrix spike solution is used.

11.5.3 Rinse a gas-tight syringe with organic-free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Sections 8.5 or 8.6) to the syringe. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 1 µL will be added to the water in the syringe.

11.6. Aqueous and low-level soil sample analysis (Purge and Trap units that sample directly from the VOA vial)

11.6.1 Units which sample from the VOA vial must be equipped with a module which automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

11.6.2 If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise, the internal and surrogate standards must be added to the vial. *Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to be analyzed as soils.

11.6.3 Soil samples, which are preserved with sodium bisulfate, must be quantitated against a curve prepared with standards containing about the same amount of sodium bisulfate as the samples (1 g in 5 mL).

11.6.4 Soil samples, which are preserved by freezing, must be allowed to thaw completely before sample analysis begins.

11.6.5 Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.7 Water Samples Not Directly Sampled from VOA Vials

11.7.1. All samples and standard solutions must be at ambient temperature before analysis.

11.7.2. Fill a syringe with the sample. If a dilution is necessary it may be made in the syringe if the sample aliquot is $\geq$ 5 µL. Check and document the pH of the remaining sample.

11.7.3. Add 50 ng of each internal and surrogate standard. The internal standards and the surrogate standards may be mixed and added as one spiking solution (this results in a 10 µg/L solution for a 5 mL sample). Inject the sample into the purging chamber. The internal and surrogate standards can be added automatically by the autosampler.

11.7.3.1. For TCLP samples, use 1 mL of TCLP leachate with 4 mL reagent water. (Note: TCLP reporting limits will be five times higher than the corresponding aqueous limits.)

11.7.4. Purge the sample for 11 minutes (trap must be below 35°C).

11.7.5. After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for approximately 3-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.7.6. Desorb and bake time and temperature are optimized for the type of trap in use. The same conditions must be used for samples and standards.

11.8. Low-Level Solids Analysis using discrete autosamplers, Methods 8260A and 5030A

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated soil/sediment sample mixed with reagent water containing the surrogates and internal standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.8.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.8.2. Weigh out 5g (or other appropriate aliquot) of sample into a 40 mL vial. Record the weight to the nearest 0.1g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 0.5g. If the sample is contaminated with analytes such that a purge amount less than 0.5g is appropriate, use the medium level method. For the medium level method, add 5g soil to 5 mL methanol containing the surrogates, mix for two minutes, allow to settle, then remove a portion of the methanol, and store in a clean Teflon®-capped vial at 4 °C until analysis. Analyze as described in Section 11.5.

11.8.3. Add 5 mL of organic free water to the VOA vial. Add surrogate/internal standard (and matrix spike solutions if required.). Add directly to the sample from Section 11.5.1.

11.8.4. The above steps must be performed rapidly and without interruption to avoid loss of volatile organics.

11.9 Medium-Level Soil/Sediment and Waste Samples

11.9.1. Sediments/soils and waste that are insoluble in methanol.

11.9.1.1 Weigh 5 g (wet weight) into a tared vial. Use a top-loading balance. Record the weight to 0.1 gram. Do not discard any supernatant liquids.

11.9.1.2 Quickly add 5 mL of methanol, and 5µL of 2500 µg/mL surrogate spiking solution to bring the final volume of methanol to 5 mL. For an LCS or MS/MSD sample, add 4.9 mL of methanol, 5µL of surrogate spike solution, and 0.1 mL of matrix spike solution. Cap the vial and shake or vortex to mix thoroughly.

Note: Sections 11.9.1.1 and 11.9.1.2 must be performed rapidly and without interruption to avoid the loss of volatile organics.

11.10. Initial review and corrective actions

11.10.1. If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minutes from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Re-analysis of samples analyzed while the system was malfunctioning is required.

11.10.2. If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Re-analysis of samples analyzed while the system was malfunctioning is required. Re-analysis must be undiluted if matrix interference is not observed.

11.10.2.1. Any samples that do not meet the internal standard criteria for the continuing calibration must be evaluated for validity. If the change in sensitivity is a matrix effect, the sample is re-analyzed to confirm. If the change in sensitivity is due to instrumental problems, all affected samples must be re-analyzed after the problem is corrected. For Ohio VAP projects, the laboratory will re-analyze any sample where the internal standard fails, and there is no evidence of matrix interference. If there is no matrix interference, the sample must be reanalyzed at the original dilution. If the internal standard is within criteria, report the second analysis. If the internal standard is still outside of criteria, the sample must be analyzed at a second dilution. If the internal standard still does not meet criteria, the sample must be diluted until the internal standard meets criteria. Multiple runs may be required.

11.11. Dilutions

11.11.1 If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution must be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be re-analyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.11.2 Guidance for Dilutions Due to Matrix

11.11.2.1 If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample

must be re-analyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement.

11.11.3 Reporting Dilutions

11.11.3.1 The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative Identification

12.1.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component, and (2) correspondence of the sample component and the standard component characteristic ions. See Table 12 for a list of the characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

12.1.1.1 The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same 12 hours as the sample.

12.1.1.2 The relative intensities of ions must agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)

12.1.2 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst must report that identification and proceed with quantitation.

12.2. Tentatively Identified Compounds (TICs)

12.2.1. If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. Guidelines are:

12.2.1.1. Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) must be present in the sample spectrum.

12.2.1.2. The relative intensities of the major ions must agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

12.2.1.3. Molecular ions present in the reference spectrum must be present in the sample spectrum.

12.2.1.4. Ions present in the sample spectrum but not in the reference spectrum must be reviewed for possible background contamination or presence of coeluting compounds.

12.2.1.5. Ions present in the reference spectrum but not in the sample spectrum must be reviewed for possible subtraction from the spectrum because of background contamination or coeluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

12.2.1.6. Computer-generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches must the analyst assign a tentative identification.

12.3. Calculations

12.3.1. Response Factor (RF)

Equation 1

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Concentration of the specific internal standard, ng

C_x = Concentration of the compound being measured, ng

12.3.2. Standard Deviation (SD)

Equation 2

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(Xi - X)^2}{N - 1}}$$

Where:

X_i = Value of X at i through N

N = Number of points

X = Average value of X_I

12.3.3. Percent Relative Standard Deviation (%RSD)

Equation 3

$$\%RSD = \frac{\textbf{Standard Deviation}}{\overline{RF_i}} \times 100$$

$\overline{RF_i}$ = **Mean of RF values in the curve**

12.3.4. Percent Drift Between the Initial Calibration and the Continuing Calibration

Equation 4

$$\% \text{ Drift} = \frac{C_{expecte} - C_{found}}{C_{expecte}} \times 100$$

Where:

$C_{expecte}$ = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.3.5. Target Compound and Surrogate Concentrations

12.3.5.1 Concentrations in the sample may be determined from linear or second order (quadratic) curve fitted to the initial calibration points, or from the average

response factor of the initial calibration points. Average response factor may only be used when the % RSD of the response factors in the initial calibration is ≤ 15%.

12.3.5.2 Calculation of Concentration Using Average Response Factors

Equation 5

$$\text{Concentration } \mu g / L = \frac{x}{RF}$$

12.3.5.3 Calculation of Concentration using Linear Fit

Equation 6

$$\text{Concentration } \mu g / L = A + Bx$$

12.3.5.4. Calculation of Concentration Using Quadratic Fit

Equation 7

$$\text{Concentration } \mu g / L = A + Bx + Cx^2$$

Where:

x is defined in Equations 8, 9, and 10

A is a constant defined by the intercept

B is the slope of the curve

C is the curvature

12.3.5.5. Calculation of ***x*** for Water and water-miscible waste:

Equation 8

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

X = ug/L

A_x = Area of characteristicion for the compoundbeing measured (secondaryion quantitationis allowedonly when there are sampleinterferenceswith the primaryion)

A_{is} = Area of the characteristicion for the internalstandard

I_s = Amountof internalstandardaddedin ng

$$\text{Dilution Factor} = D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

V_o = Volumeof waterpurged,mL

12.3.5.6. Calculationof x for Mediumlevelsoils:

Equation 9

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

X = ug/kg

A_x, I_s, D_f, A_{is}, same as for water

V_t = Volumeof totalextract,mL

V_a = Volumeof extractaddedfor purging,µL

W_s = Weight of sample extracted, g

$$D = \frac{100 - \%\,\text{moisture}}{100}$$

12.3.5.7. Calculationof x for Low levelsoils:

Equation 10

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

X = ug/kg

A_x, I_s, A_{is}, same as for water

D = as for medium level soils

W_s = Weight of sample added to the purge vessel, g

12.3.5.8. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area in the total ion chromatogram for the compound being measured

A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

In other words, the concentration is equal to *x* as defined in Equations 8, 9, and 10.

12.3.6. MS/MSD Recovery

Equation 11

$$\text{Matrix Spike Recovery, \%} = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spike Sample result

SR = Sample Result

SA = Spiked amount

12.3.7. Relative % Difference calculation for the MS/MSD:

Equation 12

$$\text{RPD} = \frac{|\text{MSR - MSDR}|}{\frac{1}{2}(\text{MSR} + \text{MSDR})} \times 100$$

Where:

RPD = Relative percent difference
MSR = Matrix spike result
MSDR = Matrix spike duplicate result

12.4 Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002.

13. METHOD PERFORMANCE

13.1. Method Detection Limit

13.1.1. Generally, each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is defined in QA SOPs NC-QA-021 and CA-Q-S-006. When non-standard compounds are analyzed at client request, lesser requirements are possible with client agreement. At a minimum, a standard at the reporting limit must be analyzed to demonstrate the capability of the method. The non-standard compound must be detected in the reporting limit standard to be acceptable.

13.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of a standard at the reporting limit and a single point calibration.

13.2. Initial Demonstration

13.2.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.3. Training Qualification

13.3.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.3.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by annual refresher training.

15.2. All waste will be disposed of in accordance with Federal, State, and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.3. The following waste streams are produced when this method is carried out.

15.3.1. **Acidic material from the auto-sampler**: Waste stream must be collected and neutralized before discharge to a sewer system if the pH is less than 5.

15.3.2. **Methanol waste from rinses and standards:** Methanol waste is discarded as a flammable liquid in a solvent waste container identified as "Flammable Liquid Waste".

15.3.3. **All samples including purged and extracted soils and waters:** Samples are collected in boxes and removed from the lab to storage. The Waste Coordinator handles crushing the vials and proper disposal.

15.3.4. **Solid samples** - Stirbars are removed from the sample. The contents of the vial are poured into a beaker, and the soil allowed to settle out. The soil is disposed of in the solid waste container.

16. REFERENCES

16.1. References

16.1.1. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260B, Update III, December 1996

16.1.2. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260A, Update II, September 1994

16.1.3. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Purge-and-Trap for Aqueous Samples, Method 5030B, Rev 2, December 1996

16.1.4. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Purge-and-Trap for Aqueous Samples, Method 5030A, Rev 1, July 1992

16.1.5. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Method 5035, Rev 0, December 1996

16.1.6 SW846, Test Methods for Evaluating Solid Waste Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Method 5035A, Draft Revision 1, July 2002

16.1.7 SW846, Test Methods for Evaluation Solid Waste, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Method 8260C, Revision 3, August 2006.

16.1.7 TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.8 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.9 Corporate Quality Management Plan (CQMP), current version

16.1.10 RevisionHistory

Historical File:		Revision 2.0: 12/15/97		Revision 0: 06/30/08 (NC-MS-019)
(formerly CORP-MS-0002NC)		Revision 2.1: 03/06/00		Revision1: 01/07/09
		Revision 2.2: 11/28/00		
		Revision 2.3: 05/23/01		
		Revision 2.4: 09/27/04		
		Revision 2.5: 04/03/07		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. GlasswareWashing,NC-QA-014

16.2.3. StatisticalEvaluationof Data and Developmentof ControlCharts,NC-QA-018

16.2.4. MethodDetectionLimitsand InstrumentDetectionLimitsNC-QA-021 and CA-Q-S-006

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-016

16.2.6. Standards and Reagents, NC-QA-017

16.2.7. LaboratoryHoldingBlanks,NC-QA-020

16.2.8. Selectionof CalibrationPoints,CA-T-P-002

16.2.9. CalibrationCurves(General),CA-Q-S-005

16.2.10. Acceptable Manual Integration Practices, CA-Q-S-002

17. MISCELLANEOUS

17.1. Modificationsfrom the referencemethod

17.1.1. A retentiontime windowof 0.2 minutesis used for all components,since some data systemsdo not have the capabilityof usingthe relativeretentiontime unitsspecifiedin the reference method.

17.1.2. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW846 in order to improve the reliability of qualitative identification.

17.2 The following are protocols that must be followed to achieve the lower reporting limits required when analyzing Michigan projects.

17.2.1 Modify Sections 8.5.4 and 8.6.8 (add 5 uL of 2500 ug/mL surrogate solution for a nominal 10g sample).

17.2.2 Modify Sections 8.5.5 and 8.6.9 (add 100 uL of 50 ug/mL spike solution for a nominal 10g sample).

17.2.3 Modify Sections 8.5.6 and 8.6.10 (add 100 uL of 50 ug/mL spike solution for a nominal 10g sample).

17.2.4 Michigan reporting limits for methanol preserved soils are achieved by injecting 100 uL of the methanol extract in a 5 mL purge. The instrument is calibrated using the recommended calibration range for water of 0.5 ug/L to 100 ug/L. Some analytes are prepared at higher concentrations.

Table 1 - TestAmerica Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	Low Level 5 mL water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
Dichlorodifluoromethane	75-71-8	5	1	5	250	250
Chloromethane	74-87-3	5	1	5	250	250
Bromomethane	74-83-9	5	1	5	250	250
Vinyl chloride	75-01-4	5	1	5	250	250
Chloroethane	75-00-3	5	1	5	250	250
Trichlorofluoromethane	75-69-4	5	1	5	250	250
Acrolein	107-02-8	100	20	100	5,000	5,000
Acetone	67-64-1	20	10	20	1,000	1,000
Trichlorotrifluoroethane	76-13-1	5	1	5	250	250
Iodomethane	74-88-4	5	1	5	250	250
Carbon disulfide	75-15-0	5	1	5	250	250
Methylene chloride	75-09-2	5	1	5	250	250
tert-Butyl alcohol	75-65-0	200	50	200	10,000	10,000
1,1-Dichloroethene	75-35-4	5	1	5	250	250
1,1-Dichloroethane	75-34-3	5	1	5	250	250
Trans-1,2-Dichloroethene	156-60-5	5.0	1.0	5.0	250	250
Acrylonitrile	107-13-1	100	20	100	5,000	5,000
Methyl *tert*-butyl ether (MTBE)	1634-04-4	5	1	5	250	250
Hexane	110-54-3	5	1	5	250	250
cis-1,2-Dichloroethene	156-59-2	5	1	5	250	250
1,2-Dichloroethene (Total)	540-59-0	10	2	10	500	500
Tetrahydrofuran	109-99-9	20	5	20	1,000	1,000
Chloroform	67-66-3	5	1	5	250	250
1,2-Dichloroethane	107-06-2	5	1	5	250	250
Dibromomethane	74-95-3	5	1	5	250	250
2-Butanone	78-93-3	20	5	20	1,000	1,000
1,4-Dioxane	123-91-1	500	200	500	25,000	25,000
1,1,1-Trichloroethane	71-55-6	5	1	5	250	250
Carbon tetrachloride	56-23-5	5	1	5	250	250
Bromodichloromethane	75-27-4	5	1	5	250	250
1,2-Dichloropropane	78-87-5	5	1	5	250	250
cis-1,3-Dichloropropene	10061-01-5	5	1	5	250	250
Trichloroethene	79-01-6	5	1	5	250	250
Dibromochloromethane	124-48-1	5	1	5	250	250
1,2-Dibromoethane	106-93-4	5	1	5	250	250
1,2,3-Trichloropropane	96-18-4	5	1	5	250	250

Table 1 - TestAmerica Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	Low Level 5 mL water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
1,1,2-Trichloroethane	79-00-5	5	1	5	250	250
Benzene	71-43-2	5	1	5	250	250
Ethylmethacrylate	97-63-2	5	1	5	250	250
Trans-1,3-Dichloropropene	10061-02-6	5	1	5	250	250
Bromoform	75-25-2	5	1	5	250	250
4-Methyl-2-pentanone	108-10-1	20	5	20	1000	1,000
2-Hexanone	591-78-6	20	5	20	1000	1,000
Tetrachloroethene	127-18-4	5	1	5	250	250
Toluene	108-88-3	5	1	5	250	250
1,1,2,2-Tetrachloræthane	79-34-5	5	1	5	250	250
2-Chloroethyl vinyl ether	110-75-8	N/A[2]	N/A	50	1000	1,000
Vinyl acetate	108-05-4	10	2	10	500	500
Chlorobenzene	108-90-7	5	1	5	250	250
Ethylbenzene	100-41-4	5	1	5	250	250
Styrene	100-42-5	5	1	5	250	250
t-1,4-Dichloro-2-butene	110-57-6	5	1	5	250	250
m and p Xylenes		10	2	10	500	500
o-xylene	95-47-6	5.0	1	5	250	250
Total xylenes	1330-20-7	10	2	10	500	500
1,3-Dichlorobenzene	541-73-1	5	1	5	250	250
1,4-Dichlorobenzene	106-46-7	5	1	5	250	250
1,2-Dichlorobenzene	95-50-1	5	1	5	250	250
2,2-Dichloropropane	590-20-7	5	1	5	250	250
Bromochloromethane	74-97-5	5	1	5	250	250
1,1-Dichloropropene	563-58-6	5	1	5	250	250
Bromodichloromethane	75-27-4	5	1	5	250	250
1,2-Dichloropropane	78-87-5	5	1	5	250	250
1,3-Dichloropropane	142-28-9	5	1	5	250	250
Isopropylbenzene	98-82-8	5	1	5	250	250
Bromobenzene	108-86-1	5	1	5	250	250
n-Propylbenzene	103-65-1	5	1	5	250	250
2-Chlorotoluene	95-49-8	5	1	5	250	250
4-Chlorotoluene	106-43-4	5	1	5	250	250
1,3,5-Trimethylbenzene	108-67-8	5	1	5	250	250
Tert-Butylbenzene	98-06-6	5	1	5	250	250
1,2,4-Trimethylbenzene	95-63-6	5	1	5	250	250
Sec-butylbenzene	135-98-8	5	1	5	250	250

Table 1 - TestAmerica Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	Low Level 5 mL water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
4-Isopropyltoluene	99-87-6	5	1	5	250	250
n-Butylbenzene	104-51-8	5	1	5	250	250
1,2,4-Trichlorobenzene	120-82-1	5	1	5	250	250
Napthalene	91-20-3	5	1	5	250	250
Hexachlorobutadiene	87-68-3	5	1	5	250	250
1,2,3-Trichlorobenzene	87-61-6	5	1	5	250	250
Acetonitrile	75-05-8	100	20	100	5000	500
Cyclohexane	110-82-7	10	1	10	500	500
Methyl Acetate	79-20-9	10	10	10	500	500
Methyl cyclohexane	108-87-2	10	1	10	500	500

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

[2] 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

Table 2 - TestAmerica Primary Standard Calibration Levels, 5 mL purge Solid

Compound	Calibration Level ug/kg				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2-Dichloroethane-d4 (Surrogate)	5	20	50	100	200
Toluene-d8 (Surrogate)	5	20	50	100	200
4-Bromofluorobenzene (Surrogate)	5	20	50	100	200
Dichlorodifluoromethane	5	20	50	100	200
Chloromethane	5	20	50	100	200
Bromomethane	5	20	50	100	200
Vinyl chloride	5	20	50	100	200
Chloroethane	5	20	50	100	200
Trichlorofluoromethane	5	20	50	100	200
Acrolein	50	200	500	1000	2000
Acetone	5	20	50	100	200
Trichlorotrifluoroethane	5	20	50	100	200
Iodomethane	5	20	50	100	200
Carbon disulfide	5	20	50	100	200
Methylene chloride	5	20	50	100	200
tert-Butyl alcohol	100	400	1,000	2,000	4,000
1,1-Dichloroethene	5	20	50	100	200

Table 2 - TestAmerica Primary Standard Calibration Levels, 5 mL purge Solid

Compound	Calibration Level ug/kg				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,1-Dichloroethane	5	20	50	100	200
trans-1,2-Dichloroethene	5	20	50	100	200
Acrylonitrile	10	40	100	200	400
Methyl *tert*-butyl ether (MTBE)	5	20	50	100	200
Hexane	5	20	50	100	200
cis-1,2-Dichloroethene	5	20	50	100	200
Tetrahydrofuran	5	20	50	100	200
Chloroform	5	20	50	100	200
1,2-Dichloroethane	5	20	50	100	200
Dibromomethane	5	20	50	100	200
2-Butanone	5	20	50	100	200
1,4-Dioxane	250	1000	2,500	5,000	10,000
1,1,1-Trichloroethane	5	20	50	100	200
Carbon tetrachloride	5	20	50	100	200
Bromodichloromethane	5	20	50	100	200
1,2-Dichloropropane	5	20	50	100	200
cis-1,3-Dichloropropene	5	20	50	100	200
Trichloroethene	5	20	50	100	200
Dibromochloromethane	5	20	50	100	200
1,2-Dibromoethane	5	20	50	100	200
1,2,3-Trichloropropane	5	20	50	100	200
Acetonitrile	50	200	500	1000	2000
1,1,2-Trichloroethane	5	20	50	100	200
Benzene	5	20	50	100	200
Ethylmethacrylate	5	20	50	100	200
trans-1,3-Dichloropropene	5	20	50	100	200
Bromoform	5	20	50	100	200
4-Methyl-2-pentanone	5	20	50	100	200
2-Hexanone	5	20	50	100	200
Tetrachloroethene	5	20	50	100	200
Toluene	5	20	50	100	200
1,1,2,2-Tetrachloroethane	5	20	50	100	200
2-Chloroethyl vinyl ether	10	40	100	200	400
Vinyl acetate	5	20	50	100	200
Chlorobenzene	5	20	50	100	200
Ethylbenzene	5	20	50	100	200
Styrene	5	20	50	100	200
t-1,4-Dichloro-2-butene	5	20	50	100	200
m and p Xylenes	10	40	100	200	400
o-xylene	5	20	50	100	200

Table 2 - TestAmerica Primary Standard Calibration Levels, 5 mL purge Solid

Compound	Calibration Level ug/kg				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,3-Dichlorobenzene	5	20	50	100	200
1,4-Dichlorobenzene	5	20	50	100	200
1,2-Dichlorobenzene	5	20	50	100	200
2,2-Dichloropropane	5	20	50	100	200
Bromochloromethane	5	20	50	100	200
1,1-Dichloropropene	5	20	50	100	200
Bromodichloromethane	5	20	50	100	200
1,2-Dichloropropane	5	20	50	100	200
1,3-Dichloropropane	5	20	50	100	200
Isopropylbenzene	5	20	50	100	200
Bromobenzene	5	20	50	100	200
n-Propylbenzene	5	20	50	100	200
2-Chlorotoluene	5	20	50	100	200
4-Chlorotoluene	5	20	50	100	200
1,3,5-Trimethylbenzene	5	20	50	100	200
tert-Butylbenzene	5	20	50	100	200
1,2,4-Trimethylbenzene	5	20	50	100	200
sec-butylbenzene	5	20	50	100	200
4-Isopropyltoluene	5	20	50	100	200
n-Butylbenzene	5	20	50	100	200
1,2,4-Trichlorobenzene	5	20	50	100	200
Napthalene	5	20	50	100	200
Hexachlorobutadiene	5	20	50	100	200
1,2,3-Trichlorobenzene	5	20	50	100	200

Table 2A - TestAmerica Primary Standard Calibration Levels, Low Level [1(Water)]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Dibromofluoromethane (Surrogate)	1	5	10	20	40
1,2-Dichloroethane-d4 (Surrogate)	1	5	10	20	40
Toluene-d8 (Surrogate)	1	5	10	20	40
Bromofluorobenzene (Surrogate)	1	5	10	20	40
Dichlorodifluoromethane	1	5	10	20	40
Chloromethane	1	5	10	20	40
Vinyl Chloride	1	5	10	20	40
Bromomethane	1	5	10	20	40
Chloroethane	1	5	10	20	40
Trichlorofluoromethane	1	5	10	20	40
Acrolein	10	50	100	200	400
Acetone	2	10	20	40	80
1,1-Dichloroethene	1	5	10	20	40
Trichlorotrifluoroethane	1	5	10	20	40
Iodomethane	1	5	10	20	40
Carbon Disulfide	1	5	10	20	40
Methylene Chloride	1	5	10	20	40
Acetonitrile	10	50	100	200	400
Acrylonitrile	2	10	20	40	80
Methyl tert-butyl ether	1	5	10	20	40
trans-1,2-Dichloroethene	1	5	10	20	40
Hexane	1	5	10	20	40
Vinyl acetate	1	5	10	20	40
1,1-Dichloroethane	1	5	10	20	40
tert-Butyl Alcohol	20	100	200	400	800
2-Butanone	2	10	20	40	80
cis-1,2-dichloroethene	1	5	10	20	40
2,2-Dichloropropane	1	5	10	20	40
Bromochloromethane	1	5	10	20	40
Chloroform	1	5	10	20	40
Tetrahydrofuran	1	5	10	20	40
1,1,1-Trichloroethane	1	5	10	20	40
1,1-Dichloropropene	1	5	10	20	40
Carbon Tetrachloride	1	5	10	20	40
1,2-Dichloroethane	1	5	10	20	40
Benzene	1	5	10	20	40
Trichloroethene	1	5	10	20	40
1,2-Dichloropropane	1	5	10	20	40

Table 2A - TestAmerica Primary Standard Calibration Levels, Low Level [1(Water)]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,4-Dioxane	50	250	500	1000	2000
Dibromomethane	1	5	10	20	40
Bromodichloromethane	1	5	10	20	40
2-Chloroethyl vinyl ether	2	10	20	40	80
cis-1,3-Dichloropropene	1	5	10	20	40
4-Methyl-2-pentanone	2	10	20	40	80
Toluene	1	5	10	20	40
trans-1,3-Dichloropropene	1	5	10	20	40
Ethyl Methacrylate	1	5	10	20	40
1,1,2-Trichloroethane	1	5	10	20	40
1,3-Dichloropropane	1	5	10	20	40
Tetrachloroethene	1	5	10	20	40
2-Hexanone	2	10	20	40	80
Dibromochloromethane	1	5	10	20	40
1,2-Dibromoethane	1	5	10	20	40
Chlorobenzene	1	5	10	20	40
1,1,1,2-Tetrachloroethane	1	5	10	20	40
Ethylbenzene	1	5	10	20	40
m + p-Xylene	2	10	20	40	80
Xylene-o	1	5	10	20	40
Styrene	1	5	10	20	40
Bromoform	1	5	10	20	40
Isopropylbenzene	1	5	10	20	40
1,1,2,2-Tetrachloroethane	1	5	10	20	40
1,4-Dichloro-2-butene	1	5	10	20	40
1,2,3-Trichloropropane	1	5	10	20	40
Bromobenzene	1	5	10	20	40
n-Propylbenzene	1	5	10	20	40
2-Chlorotoluene	1	5	10	20	40
1,3,5-Trimethylbenzene	1	5	10	20	40
4-Chlorotoluene	1	5	10	20	40
tert-Butylbenzene	1	5	10	20	40
1,2,4-Trimethylbenzene	1	5	10	20	40
sec-Butylbenzene	1	5	10	20	40
4-Isopropyltoluene	1	5	10	20	40
1,3-Dichlorobenzene	1	5	10	20	40
1,4-Dichlorobenzene	1	5	10	20	40
n-Butylbenzene	1	5	10	20	40
1,2-Dichlorobenzene	1	5	10	20	40
1,2-Dibromo-3-chloropropane	1	5	10	20	40

Table 2A - TestAmerica Primary Standard Calibration Levels, Low Level [1(Water)]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2,4-Trichlorobenzene	1	5	10	20	40
Hexachlorobutadiene	1	5	10	20	40
Naphthalene	1	5	10	20	40
1,2,3-Trichlorobenzene	1	5	10	20	40
Cyclohexane	1	5	10	20	40
Methyl Acetate	2	10	20	40	80
Methylcyclohexane	1	5	10	20	40
1,3,5-Trichlorobenzene	1	5	10	20	40

[1] 25 mL purge samples analyzed at 5 mL purge on more sensitive equipment.

Table 3 - TestAmerica Appendix IX Standard and Reporting Limits, 5 mL purge

Compound	CAS Number	Reporting Limits			
		5 mL Water µg/L	Low Level 5mL purge water µg/L	Low Soil µg/kg	Medium Soil µg/mL
Allyl Chloride	107-05-1	10	2	10	500
Dichlorofluoromethane	75-43-4	10	2	10	500
Isopropyl ether	108-20-3	10	2	10	500
Chloroprene	126-99-8	5	2	5	250
n-Butanol	71-36-3	200	50	200	10,000
Propionitrile	107-12-0	20	4	20	1000
Methacrylonitrile	126-98-7	5	2	5	250
Isobutanol	78-83-1	200	50	200	10,000
Methyl methacrylate	80-62-6	5	2	5	250
1,1,1,2-Tetrachloroethane	630-20-6	5	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	10	2	10	500
Ethyl ether	60-29-7	10	2	10	500
Ethyl Acetate	141-78-6	20	4	20	1,000
2-Nitropropane	79-46-9	10	4	10	500
Cyclohexanone	108-94-1	50	20	50	2500
Isopropylbenzene	98-82-8	5	1	5	250
2-Methylnaphthalene (Michigan only)	91-57-6	NA	5	10	330

Table 4

Recommended TestAmerica Appendix IX Standard Calibration Levels, µg/L

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Allyl Chloride	5	20	50	100	200
Dichlorofluoromethane	5	20	50	100	200
Isopropyl ether	5	20	50	100	200
Chloroprene	5	20	50	100	200
n-Butanol	100	400	1,000	2,000	4,000
Propionitrile	10	40	100	200	400
Methacrylonitrile	5	20	50	100	200
Isobutanol	100	400	1,000	2,000	4,000
Methyl methacrylate	5	20	50	100	200
1,1,1,2-Tetrachloroethane	5	20	50	100	200
1,2-Dibromo-3-chloropropane	10	40	100	200	400
Ethyl ether	5	20	50	100	200
Ethyl Acetate	10	40	100	200	400
2-Nitropropane	10	40	100	200	400
Cyclohexanone	50	200	500	1,000	2,000
2-Methylnaphthalene (Michigan only)	2	10	20	40	80
Ethyl tert-butyl ether	5	20	50	100	200
tert-Amyl methyl ether	5	20	50	100	200
1,2,3-Trimethylbenzene	5	20	50	100	200

Table 5 - Internal Standards

Compound	Standard Concentration µg/mL (may vary per matrix)	Quantitation ion
Fluorobenzene	50 – 250	96
Chlorobenzene-d5	50 – 250	117
1,4-Dichlorobenzene-d4	50 – 250	152

Notes:

1) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 6 - Surrogate Standards

Surrogate Compounds	Standard Concentration µg/mL (may vary per matrix)
1,2-Dichloroethane-d_4	50 – 250
Dibromofluoromethane (not required for Method 8260C)	50 – 250
Toluene-d_8	50 - 250
4-Bromofluorobenzene	50 – 250

Notes:

1) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

2) Recovery limits for surrogates are g enerated from historical data and are maintained by the QA Dept.

3) There is no corrective action for Dibromofluoromethane for Method 8260C.

Table 7 - Matrix Spike / LCS Compounds

Compound	Standard Concentration µg /mL
1,1,1-Trichloroethane	50 – 250
1,1,2,2-Tetrachloroethane	50
1,1,2-Trichloro-1,2,2-trifluoroethane	50
1,1,2-Trichloroethane	50
1,1-Dichloroethane	50
1,1-Dichloroethene	50
1,1-Dichloropropene	50
1,2,3-Trichlorobenzene	50
1,2,3-Trichloropropane	50
1,2,4-Trichlorobenzene	50
1,2,4-Trimethylbenzene	50
1,2-Dibromo-3-chloropropane	50
1,2-Dibromoethane	50
1,2-Dichlorobenzene	50
1,2-Dichloroethane	50
1,2-Dichloroethene (total)	100
1,2-Dichloropropane	50
1,3,5-Trimethylbenzene	50
1,3-Dichlorobenzene	50
1,3-Dichloropropane	50
1,4-Dichlorobenzene	50
2,2-Dichloropropane	50
2-Butanone	50
2-Chloroethyl Vinyl Ether	100 – 500
2-Chlorotoluene	50
2-Hexanone	50
4-Chlorotoluene	50
4-Methyl-2-pentanone	50
Acetone	50
Acetonitrile	500 – 2500
Acrolein	500
Acrylonitrile	100 – 500
Benzene	50
Bromobenzene	50
Bromochloromethane	50
Bromodichloromethane	50
Bromoform	50
Bromomethane	50
Carbon disulfide	50
Carbon tetrachloride	50
Chlorobenzene	50
Chloroethane	50

Table 7 - Matrix Spike / LCS Compounds

Compound	Standard Concentration µg /mL
Chloroform	50
Chloromethane	50
cis-1,2-Dichloroethene	50
cis-1,3-Dichloropropene	50
Cyclohexane	50
Dibromochloromethane	50
Dibromomethane	50
Dichlorodifluoromethane	50
Ethylbenzene	50
Hexachlorobutadiene	50
Iodomethane	50
Isopropylbenzene	50
Isopropylether	50
Methyl acetate	50
Methyl tert-butyl ether (MTBE)	50
Methylcyclohexane	50
Methylene chloride	50
Naphthalene	50
n-Butylbenzene	50
n-Hexane (Ohio VAP only)	50
n-Propylbenzene	50
p-Isopropyltoluene	50
sec-Butylbenzene	50
Styrene	50
tert-Butylbenzene	50
Tetrachloroethene	50
Toluene	50
trans-1,2-Dichloroethene	50
trans-1,2-Dichloroethene	50
trans-1,3-Dichloropropene	50
Trichloroethene	50
Trichlorofluoromethane	50
Vinyl Acetate	50
Vinyl chloride	50
Xylenes (total)	150 – 750

Notes: 1) 5 µL of the standard is added to the LCS or matrix spiked sample. This results in a concentration of each spike analyte in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Recovery and precision limits for LCS and MS/MSD are generated from historical data and are maintained by QA Dept.

Table 8 - BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

Table 9 - SPCC Compounds and Minimum Response Factors for Method 8260B

Compound	Methods 8260B and 8260A Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	0.100
1,1,2,2-Tetrachlorœthane	0.300
Chlorobenzene	0.300

Table 10 - Method 8260C. Recommended Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification		
Volatile Compound	**Minimum Response Factor**	**Typical Response Factor**
Dichlorodifluoromethane	0.100	0.327
Chloromethane	0.100	0.537
Vinyl chloride	0.100	0.451
Bromomethane	0.100	0.255
Chloroethane	0.100	0.254
Trichlorofluoromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100	0.302
Acetone	0.100	0.151
Carbon disulfide	0.100	1.163
Methyl Acetate	0.100	0.302
Methylene chloride	0.100	0.380
trans-1,2-Dichloroethene	0.100	0.351
cis-1,2-dichloroethene	0.100	0.376
Methyl tert-Butyl Ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.100	0.216
Chloroform	0.200	0.557
1,1,1-Trichloroethane	0.100	0.442
Cyclohexane	0.100	0.579
Carbon tetrachloride	0.100	0.353
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.200	0.338
Methylcyclohexane	0.100	0.501
1,2-Dichloropropane	0.100	0.382
Bromodichloromethane	0.200	0.424
cis-1,3-Dichloropropene	0.200	0.537
trans-1,3-Dichloropropene	0.100	0.515
4-Methyl-2-pentanone	0.100	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.100	0.536
Dibromochloromethane	0.100	0.652
Styrene	0.300	1.916
Bromoform	0.100	0.413
Isopropylbenzene	0.100	2.271
1,1,2,2-Tetrachloroethane	0.300	0.782
1,3-Dichlorobenzene	0.600	1.408

Table 10 - Method 8260C: Recommended Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification (cont'd)		
Volatile Compound	**Minimum Response Factor**	**Typical Response Factor**
1,4-Dichlorobenzene	0.500	1.427
1,2-Dichlorobenzene	0.400	1.332
1,2-Dibromo-3-chloropropane	0.050	0.129
1,2,4-Trichlorobenzene	0.200	0.806

Table 11 - CCC Compounds for Method 8260B

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	30	20
1,1-Dichloroethene	30	20
Chloroform	30	20
1,2-Dichloropropane	30	20
Toluene	30	20
Ethylbenzene	30	20
n-Hexane (Ohio VAP only)	30	20

Table 12 - Characteristic Ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d_4 (Surrogate)	65	102	
Dichlorodifluoromethane	85	87	50, 101,103
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane	142	127	141
Carbon disulfide	76	78	
Trichlorotrifluoroethane	151	101	153
Acetone	43	58	
Methylene chloride	84	49	51, 86

Table 12 - Characteristic Ions

Compound	Primary*	Secondary	Tertiary
tert-Butyl alcohol	59	74	
trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl *tert* butyl ether	73		
Hexane	57	43	
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	
Tetrahydrofuran	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	
Vinyl acetate	43	86	
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121
Benzene	78	52	77
Trichloroethene	130	95	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Toluene-d_8 (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65
4-Methyl-2-pentanone	43	58	57, 100
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77
Ethylbenzene	106	91	
Xylenes	106	91	
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133

Table 12 - Characteristic Ions

Compound	Primary*	Secondary	Tertiary
Allyl Chloride	76	41	78
Acetonitrile	40	41	
Dichlorofluoromethane	67	69	
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	
Cyclohexane	56	69	84
Methyl Acetate	43	74	
Methyl cyclohexane	83	55	98

* The primary ion must be used for quantitation unless interferences are present, in which case a secondary ion may be used.

** m/z 43 may be used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Canton

SOP No. NC-MS-018, Rev. 3
Effective Date: 04/25/13

Title: GC/MS ANALYSIS BASED ON METHODS 8270C AND 8270D

[Method: SW846 8270C and 8270D]

Approvals (Signature/Date):

Tom Hula	04/24/13	[redacted]	04/24/13
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Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP NC-MS-018, Rev 2, dated 10/26/10

Facility Distribution No. _____ **Distributed To: _____**

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LIST OF TABLES AND APPENDICIES

1. SCOPE AND APPLICATION

1.1 This method is based upon SW846 8270C and 8270D, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices. Direct injection of a sample may be used in limited applications. Refer to Tables 1 and 2 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. Additional compounds may be amenable to this method. If non-standard analytes are required, they must be validated by the procedures described in Section 13 before sample analysis.

1.2 The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- Hexachlorophene is not amenable to analysis by this method.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.

1.3 The standard reporting limit of this method for determining an individual compound is approximately 0.33 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Tables 1 and 2 for specific reporting limits. Reporting limits will be proportionately higher for sample extracts that require dilution.

2. SUMMARY OF METHOD

2.1 Aqueous samples are extracted with methylene chloride using a separatory funnel and/or a continuous extractor. Solid samples are extracted with methylene chloride / acetone using sonication, or soxhlet. The extract is dried, concentrated to a final volume of 2 mL for waters and soils, and analyzed by GC/MS. Extraction procedures are detailed in SOPs NC-OP-037, NC-OP-038, NC-OP-039, NC-OP-040, and NC-OP-042.

2.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extracted into a gas chromatograph equipped with a narrow-bore fused silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

2.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. Identification of target analytes is accomplished by comparing

their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation_ ion relative to an internal standard using an appropriate calibration curve for the intended application.

3. DEFINITIONS

3.1 Refer to the TestAmerica Canton Quality Assurance Manual (QAM), current version, for definitions of terms used in this document or to the Glossary of Acronyms at the end of this document in Appendix A.

4. INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If interference is detected, it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.2 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it must be followed by the analysis of solvent to check for cross contamination.

4.5 Phthalate contamination is commonly observed in this analysis and its occurrence must be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5. SAFETY PRECAUTIONS

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.3 Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include Benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and n-nitrosodimethylamine.

5.4 The following is a list of the materials used in this method, which have a serious or significant

hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5 It is recommended that analysts break up work tasks to avoid repetitive motion tasks, such as opening a large number of vials or containers in one time period.

5.6 Exposure to chemicals must be maintained as low as reasonably achievable. All samples with stickers that read "Caution/Use Hood!" must be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.7 The preparation of standards and reagents must be conducted in a fume hood with the sash closed as far as the operation will permit.

5.8 It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents must be conducted in a fume hood with the sash closed as far as the operations will permit.

5.9 Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.10 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Canton associate. The situation must be reported immediately to a Laboratory Supervisor and the EH&S Coordinator.

6. EQUIPMENT AND SUPPLIES

6.1 Gas Chromatograph/Mass Spectrometer System: An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column must be directly coupled to the source.

6.2 Column: 20m x 0.18mm ID, 0.36µm film thickness silicon-coated fused-silica capillary column (J &

W Scientific DB-5.625 or equivalent) Alternate columns are acceptable if they provide acceptable performance.

NOTE: For PAHs determined by Large Volume Injection, a 30m x 0.25 mm ID, 0.5 36µm film thickness fused silica capillary column (Zebron or equivalent) is used.

6.3 Mass Spectrometer: Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) that meets all of the criteria in Table 4 when the GC/MS tuning standard is injected through the GC.

6.4 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.5 Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

6.6 Syringes: 5 µL and 10 uL Hamilton Laboratory grade syringes or equivalent.

6.7 Carrier gas: Ultra high purity helium.

6.8 Autosampler vials, inserts, and caps

7. REAGENTS AND STANDARDS

7.1 A minimum five-point calibration curve is prepared. The standard preparation information is detailed in the standards and reagents module in LIMS. If a quadratic regression is used, six points must be analyzed for the calibration curve. The low point must be at or below the reporting limit. Refer to Table 10 for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration. For Ohio VAP work, the low standard must be at, or below, the reporting limit.

7.2 An Internal Standard solution is prepared by diluting a purchased standard. The standard preparation information is detailed in the standards and reagents module in LIMS. Compounds in the I.S. Mix are acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.

7.3 Surrogate Standard Spiking Solution: Prepare as indicated in the preparative methods. Preparation information is detailed in the standards and reagents module in LIMS for the Organic Prep group. See appropriate preparation SOP. Surrogate compounds and levels are listed in Table 9.

7.4 GC/MS Tuning Standard: A methylene chloride solution containing decafluorotriphenylphosphine (DFTPP) is prepared. The standard preparation information is detailed in the standards and reagents module in LIMS. Pentachlorophenol, benzidine, and DDT, must also be included in the Tuning Standard. All components are at 25 ug/mL.

7.5 The standards listed in Sections 7.1 to 7.4 must be refrigerated at $\leq$ 6°C when not in use. Refrigeration at -10°C to -20°C may be used if it can be demonstrated that analytes do not fall out of solution at this temperature. The standards must be replaced at least once a year. Additional information can be found in SOP NC-QA-017.

8. SAMPLE PRESERVATION AND STORAGE

8.1 Sample extracts are stored at 4 $\pm$ 2°C. Samples and extracts must be stored in suitable glass containers with Teflon®-lined caps. (Extracts will be stored for 30 days after invoicing.)

8.2 Water samples are extracted within seven days of sampling, and the extracts are analyzed within 40 days of extraction. Solids, sludges, and organic liquids are extracted within 14 days of sampling and the extracts are analyzed within 40 days of extraction.

9. QUALITY CONTROL

9.1 Initial Demonstration of Capability

9.1.1 For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.2 For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration. For DoD projects, the initial demonstration must include an MDL study analysis of the LCS replicates and a minimum of five-point calibration.

9.2 Control Limits

9.2.1 In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined periodically. Control limits are established by the laboratory as described in SOP NC-QA-018. Control limits are easily accessible via LIMs

9.2.2 If samples are diluted, the surrogate and matrix spike recoveries must be reported with a flag. For DoD projects, all surrogates must be within control limits.

9.2.3 All surrogate, LCS, and MS recoveries must be entered into LIMS so that accurate historical control limits can be generated.

9.2.4 Refer to the QC Program document (QA-003) for further details of control limits.

9.3 Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank (MB). Batches are defined at the sample preparation stage. Batches must be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the TestAmerica Canton QC Program document (QA-003) for further details of the batch definition.

9.4 Method Blank

9.4.1 A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water for aqueous samples and sodium sulfate for soil samples Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common lab contaminants, see below). Any method blank contamination above the reporting limit must be less than 1/10 of the measured concentration of any sample in the associated preparation batch. Refer to SOP NC-QA-016 for DoD requirements.

9.4.1.1 If the analyte is a common laboratory contaminant the data may be reported with qualifiers if the concentration of the analyte is less than five times the RL. Such action must be taken in consultation with the client.

9.4.1.2 Re-analysis of any samples with reportable concentrations of analytes found in the method blank is required unless other actions are agreed with the client.

9.4.1.3 If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank the data may be reported with qualifiers. Such action should be taken in consultation with the client. NOTE: For Ohio VAP work, there can be no target analyte greater than the RL in the method blank All samples associated with an unacceptable method blank must be re-extracted and re-analyzed. The exceptions are as follows: (a) insufficient sample for re-extraction/re-digestion, (b) expired holding times, or (c) the analytes detected in the Method Blank are non-detect in the associated samples.

9.4.2 The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the method blank and affected samples will normally be required. Consultation with the client must
take place. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted if sufficient volume of sample remains.

9.4.3 If re-analysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments must be made in a narrative to provide further documentation.

9.4.4 Refer to the TestAmerica Canton QC Program document (QA-003) for further details of the corrective actions.

9.5 Laboratory Control Sample (LCS)

9.5.1 A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All control analytes must be within established control limits. The LCS is spiked with the compounds listed in Table 6 unless specified by a client or agency.

9.5.2 If any control analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur. All non-controlling compounds must attain a recovery of 10% or greater if the compound is on the client's list. Corrective action may include re-extraction and re-analysis of the batch. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted if sufficient volume of sample remains.

9.5.2.1 If the batch is not re-extracted and re-analyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not re-analyzing might be that the matrix spike (MS) and matrix spike duplicate (MSD) are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS).

9.5.2.2 If re-extraction and re-analysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5.2.3 The LCS must have acceptable surrogate recoveries. If surrogate recoveries are low, re-extraction of the LCS and affected samples will normally be required. Consultation with the client should take place. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted. The exceptions are as follows: (a) insufficient sample for re-extraction/re-digestion, (b) expired holding times, or (c) the LCS is biased high and the samples are non-detect for those analytes.

9.5.3 Ongoing monitoring of the LCS over time provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1 A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples. The MS/MSD is spiked with the same subset of analytes as the LCS (see Table 6). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically-generated limits.

9.6.1.1 If the recovery for any component is outside QC limits for both the matrix spike / spike duplicate and the LCS the laboratory is out of control and corrective action must be taken. For client specific samples, corrective action may include re-preparation and re-analysis of the batch.

9.6.1.2 The matrix spike / matrix spike duplicate must be analyzed at the same dilution as the un-spiked sample, even if the matrix spike compounds will be diluted out.

9.7 Surrogates

9.7.1 Every sample, method blank and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 9.

9.7.2 If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

9.7.2.1 Check all calculations for error.

9.7.2.2 Ensure that instrument performance is acceptable.

9.7.2.3 Recalculate the data and/or re-analyze the extract if either of the above checks reveals a problem.

9.7.2.3.1 It is only necessary to re-prepare / re-analyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

Note: If all associated QC meets criteria (method blank (LCS)), up to one surrogate per fraction may be outside of acceptance criteria , as long as the recovery is greater than 10%. **Note:** For Ohio VAP and DoD samples, all surrogates must be within acceptance criteria. The exceptions for Ohio VAP are as follows: (a) insufficient sample for re-extraction, or (b) the surrogates are biased high and the samples are non-detect.

9.7.3 If the sample with surrogate recoveries outside the recovery limits was a sample used for a MS/MSD and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample, the MS, and the MSD do not require re-analysis as this phenomenon would indicate a possible matrix problem.

9.7.4 If the sample is re-analyzed and the surrogate recoveries in the re-analysis are acceptable, then the problem was within the analyst's control and only the re-analyzed data must be reported (unless the re-analysis was outside holding times, in which case, reporting both sets of results may be appropriate).

9.7.5 If the re-analysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effect.

9.8 Nonconformance and Corrective Action

9.8.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action.

10. CALIBRATIONAND STANDARDIZATION

10.1 Summary

10.1.1 The instrument is tuned for DFTPP, calibrated initially with a minimum five-point calibration curve, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 3.

10.1.2 For DoD work, refer to SOP NC-QA-016 for specific details.

10.2 All standards and extracts are allowed to warm to room temperature before injecting.

10.3 Instrument Tuning

10.3.1 At the beginning of every 12-hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table 4) are achieved for DFTPP (decafluorotriphenylphosphine).

10.3.2 Inject the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 4 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.3.3 The GC/MS tuning standard must also be used to evaluate the inertness of the chromatographic system. The tailing factor for benzidine must be less than 3.0. The tailing factor for pentachlorophenol must be less than 5. For Method 8270D, benzidine and pentachlorochlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2. DDT must be included in the tuning standard, and its breakdown must be < 20%. Refer to Section 12 for the appropriate calculations.

NOTE: Breakdown and trailing factor are not applicable for LVI PAHs.

10.4 Initial Calibration

10.4.1 Internal Standard Calibration Procedure. Internal standards are listed in Table 5. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

10.4.2 Compounds should be assigned to the IS with the closest retention time. Refer to Table 11 for internal standard corresponding analytes.

10.4.3 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest. Six standards must be used for a quadratic least squares calibration. Add the internal standard mixture to result in 2 ng on column. (For example, 5 uL of 80ppm IS mix is added to 100 uL of extract. This results in 4 ng; but only 0.5 uL is injected, resulting in a final on column amount of 2 ng.). The concentration ranges of all analytes are listed in Table 10. For Ohio VAP work, the low standard must be at or below the reporting limit.

10.4.4 For LVI PAH analysis, 5 uL of 8 ppm IS mix is added to 100 uL of extract. The calibration standards are diluted by a 10x factor, however 10x more is injected (5 uL injected rather than the normal 0.5 uL), keeping the calibration concentration the same as the non-LVI PAHs.

10.4.5 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Table 5 lists the analytes and characteristic ions analyzed in the laboratory. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in Section 12. For Method 8270C, verify that the SPCC and CCC criteria in Sections 10.4.5 and 10.4.7 are met. **No sample analysis must be performed unless these criteria are met.**

10.4.6 System Performance Check Compounds (SPCCs) (Method 8270C). The minimum average RF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

SPCC Compounds:

N-nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

10.4.7 Initial Calibration Criteria for Method 8270D

10.4.7.1 The RSD should be less than 20% for each analyte. For analytes that fail, use linear or quadratic curve with 0.99 correlation coefficient.

NOTE: If compliance with Method 8270C is required, the RSD limit is 15%.

10.4.7.2 No more than 10% of compounds can fail the 20%/0.99 correlation requirement.

10.4.7.3 If more than 10% of analytes fail both 20% RSD and 0.99 correlation, then recalibration is necessary.

10.4.7.4 Any individual analyte that fails both 20% RSD and 0.99 correlation must have any positive result flagged as estimated and will be noted in the narrative.

10.4.7.5 For any analyte non-detect associated with a calibration that fails the 20% RSD/0.99 correlation/minimum response factor criteria, there must be a demonstration of adequate sensitivity at the quantitation limit.

10.4.7.6 Minimum response factor should be met, especially for the low level standard.

10.4.7.7 Any individual analyte that fails the minimum response factor set in the SOP must have a demonstration of sensitivity in the analytical batch to report non-detects. The demonstration of sensitivity is analysis of a low level CCV (at or below the reporting limit). The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detects to be reported without flagging. The low level CCV would normally be analyzed immediately after the mid-level CCV. In general, Table 4 in the method should be used as guidance in setting minimum response factors in the SOP; but the RFs may be modified if appropriate (for example, if especially low-level analysis is performed).

10.4.7.8 For Method 8270D, the minimum response factors are listed in Table 12.

10.4.8 Calibration Check Compounds (CCCs) (Method 8270C). The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this criterion.

10.4.8.1 If none of the CCCs are required analytes, project-specific calibration specifications must be agreed with the client.

10.4.8.2 CCC Compounds

Phenol
Acenaphthene
1,4-Dichlorobenzene
N-nitrosodiphenylamine
2-Nitrophenol
Pentachlorophenol
2,4-Dichlorophenol
Fluoranthene
Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

10.4.8.3 Continuing Calibration Criteria for Method 8270D

10.4.8.3.1 At least 80% of analytes must have a %D less than or equal to 20%.

10.4.8.3.2 Minimum response factors must be evaluated.

10.4.9 If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst must evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve, then the appropriate curve with no forced intercept must be used for quantitation.

10.4.9.1 If an analyte in the initial calibration is > 15%, then calibration on a curve must be used. Quadratic curve fits must be used if the compound has historically

exhibited a nonlinear response. The analyst must consider instrument maintenance to improve the linearity of response. Use of 1/Concentration^2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. If Relative Standard Error (RSE) is used to evaluate the curve, it must be better than 15%. If the % RSD is >15%, the analyst may drop the low or high points in the ICAL, as long as a minimum of five points are maintained and the quantitation range is adjusted accordingly. If the % RSD is still >15%, a quadratic or linear curve must be used. The coefficient of determination (r^2) must be $\geq$ 0.990. If the coefficient of determination is < 0.990, then any hits for these compounds must be flagged as estimated. If a curve is not linear for any compound that is found in a sample, the result must be flagged as estimated. Linear is defined as <15% RSD or a coefficient of determination of 0.990.

Note: For Method 8270D, analytes using the linear calibration fit should have the read back concentration of the low level standard evaluated. The read back concentration should be within 30% of the true value. Any sample detects for analytes that fail the read back criterion and are using a linear calibration must be flagged as estimated, or described in the narrative.

Note: For Ohio VAP work, the low standard must be at or below the reporting limit.

Note: Several components do not respond well by this method (poor linearity). These compounds are indene, benzoic acid, benzaldehyde, caprolactam, 1,3,5-Trinitrobenzene, dinoseb, benzidine, alpha alpha-dimethyl phenethylamine, acrylamide, 4-Nitroquinoline-1-oxide, famphur, benzenethiol, kepone, and 2,4-Toluenediamine. If these compounds are requested by a client and hits are found, results will be flagged as estimation. Sensitivity as demonstrated by the low standard is sufficient to substantiate a non-detect.

10.4.9.1 If time remains in the 12-hour period initiated by the DFTPP injection before the initial calibration, samples must be analyzed. Otherwise, proceed to continuing calibration.

10.4.9.2 Quantitation is performed using the calibration curve or average response factor from the initial curve.

10.5 Initial Calibration Verification (ICV)

10.5.1 Calibration accuracy is verified by analyzing a second source standard (ICV) immediately after the initial calibration. The recovery CCC compounds must be $\leq$ 20%. The recovery for non-CCC compounds must be $\leq$ 50% with an allowance of up to six compounds >50%.

10.5.2 For Method 8270D, the suggested acceptance limit is 70-130% for all analytes.

10.6 Continuing Calibration

10.6.1 At the start of each 12-hour period, analyze a GC/MS tuning standard. The injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 4.

10.6.2 Following a successful DFTPP analysis the continuing calibration standard(s) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. A mid-level calibration standard is used for the continuing calibration.

10.6.3 For Method 8270C, the following criteria must be met for the continuing calibration to be acceptable:

- The SPCC compounds must have a response factor of $\geq$ 0.05.
- The percent difference or drift of the CCC compounds from the initial calibration must be $\leq$ 20% (see Section 12 for calculations). In addition, the percent difference or drift of all analytes must be $\leq$ 50%, with allowance for up to four compounds to be greater than 50%.
- The internal standard response must be within 50-200% of the response in the mid level of the initial calibration.
- The internal standard retention times must be within 30 seconds of the retention times in the mid-level of the initial calibration.

  Note: There are no internal standard criteria for samples. Criteria are only for continuing and initial calibrations.

  Note: Ohio VAP requires that any sample with internal standard outliers be re-analyzed undiluted unless a matrix effect is observed. If there is no matrix interference, the sample must be re-analyzed at the original dilution. If the internal standard is within criteria, report the second analysis. If the internal standard is still outside of criteria, the sample must be diluted until the internal standard meets criteria. Multiple runs may be required. The criteria for acceptance are between 50% and 200% of the same internal standard in continuing calibration.

10.6.3.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.6.3.2. For Method 8270D, any sample non-detects for an analyte that fails the SOP criteria low, must have a low level CCV (CCV at the RL) in the batch as a sensitivity demonstration. The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detect samples to be reported without flagging.

10.6.4. Once the above criteria have been met, sample analysis will begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis will proceed until 12 hours from the injection of the DFTPP have passed. (A sample *injected* less than 12 hours after the DFTPP is acceptable.)

11. PROCEDURE

11.1 Sample Preparation

11.1.1 Samples are prepared following SOP NC-OP-037, NC-OP-038, NC-OP-039, or NC-OP-040.

11.1.2 For DoD work, refer to SOP NC-QA-016 for specific details.

11.2 Sample Analysis Procedure

11.2.1 Calibrate the instrument as described in Section 10. Depending on the target compounds required by the client, it may be necessary to use more than one calibration standard.

11.2.2 Analyze all samples using the same instrument conditions as the preceding continuing calibration standard.

11.2.3 Add internal standard to the extract to result in 2 ng injected on column. Mix thoroughly before injection into the instrument. For LVI PAHs, the addition should result in 0.2 ng injected on column.

11.2.4 Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.

11.2.5 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in Section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.2.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system. Chromatograms before and after manual integration are required by many programs. Additional information on manual integration can be found in SOP CA-Q-S-002.

11.2.7 Target compounds identified by the data system are evaluated using the criteria listed in Section 12.1.

11.2.8 Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) must be performed if required by the client. They are evaluated using the criteria in Section 12.3.

11.3 Dilutions

11.3.1 If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution must be in the upper half of the calibration range. Samples must be screened to determine the

appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be re-analyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.3.2 Guidance for Dilutions Due to Matrix

11.3.2.1 If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be re-analyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment. For example, samples containing organic acids must be analyzed at a higher dilution to avoid destroying the column.

11.3.3 Reporting Dilutions

11.3.3.1 The most concentrated dilution with target compounds within the calibration range will be reported. Other dilutions will only be reported at client request.

11.4 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at 4 $\pm$2°C protected from light in screw cap vials equipped with unpierced Teflon®-lined septa.

11.5 Retention Time Criteria for Samples

11.5.1 If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Re-analysis of samples analyzed while the system was malfunctioning is required.

11.5.2 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure no analytes have shifted outside their retention time windows.

11.6 Procedural Variations

11.6.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo and approved by a Technical Specialist. The Nonconformance Memo must be filed in the project file. Any unauthorized deviations from this procedure must also be documented as a non-conformance with a cause and corrective action described.

11.7 Troubleshooting Guide

11.7.1 Daily Instrument Maintenance

11.7.1.1 In addition to the checks listed in the instrument maintenance schedule in the TestAmerica Canton Quality Assurance Manual (QAM), current version, the following daily maintenance must be performed.

11.7.1.1.1 Clip column as necessary.

11.7.1.1.2 Install new or cleaned injection port liner as necessary.

11.7.1.1.3 Install new septum as necessary.

11.7.1.1.4 Perform auto-tune.

11.7.2 Major Maintenance

11.7.2.1 A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the source, and replacing the multiplier. Refer to the manufacturer's manual for specific guidance.

12. DATA ANALYSIS AND CALCULATIONS

12.1 Qualitative Identification

12.1.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NBS library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

12.1.1.1 The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same 12 hours as the sample.

12.1.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

12.1.1.3 The characteristic ions of a compound must maximize in the same scan or within one scan of each other.

12.1.1.4 The relative intensities of ions must agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

12.1.2 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst must report that identification and

proceed with quantitation.

12.2 Mass chromatogram searches

12.2.1 Certain compounds are unstable in the calibration standard and cannot be calibrated in the normal way. In particular, the compound hexachlorophene (CAS 70-30-4) falls into this category, and is required for Appendix IX analysis. For this analyte, a mass chromatogram search is made.

12.2.1.1 Hexachlorophene

12.2.1.1.1 Display the mass chromatograms for mass 196 and mass 198 for the region of the chromatogram from at least 2 minutes before chrysene-d12 to at least 4 minutes after chrysene-d12. If peaks for both ions coincide, then the analyst evaluates the spectrum for the presence of hexachlorophene. No quantitation is possible.

12.3 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification must be determined by the type of analyses being conducted or by client request. Computer-generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

12.3.1 Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) must be present in the sample spectrum.

12.3.2 The relative intensities of the major ions must agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30%and 70%.)

12.3.3 Molecular ions present in the reference spectrum must be present in the sample spectrum.

12.3.4 Ions present in the sample spectrum, but not in the reference spectrum, must be reviewed for possible background contamination or presence of co-eluting compounds.

12.3.5 Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12.3.6 Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.3.7 Note: For water samples, the TIC searches begin with compounds eluting after the first surrogate (2-Fluorophenol). For solid samples, the TIC searches begin with compounds eluting after the Aldol Condensation Product. Any compounds eluting before these

analytes are considered volatile analytes are reported in the volatile analysis. A possible exception to this general rule would be if an early eluting compound were the reason for a sample dilution.

12.3.8 If a client requests 10 TICs, the laboratory supplies a minimum of 10. For a request of 20 TICS, the laboratory would supply a minimum of 20--assuming that number of compounds was available.

12.4 Anyone evaluating data is trained to know how to handle isomers with identical mass spectra and close elution times. These include:

Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Extra precautions concerning these compounds are to more closely scrutinize retention time vs. the calibration standard and also to check that all isomers have distinct retention times.

A second category of problem compounds would be the poor responders or compounds that chromatograph poorly. Included in this category would be:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol

Manually checking the integrations would be appropriate for these compounds.

12.5 Calculations

12.5.1 Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

$\overline{RF}$ = Mean of RFs from intial caibration for a compound

SD = Standard deviation of RFs from initial calibration for a compound,

$$= \sqrt{\sum_{i=1}^{N} \frac{\left(RFi - \overline{RF}\right)^2}{N-1}}$$

RFi = RF for each of the calibration levels

N = Number of RF values

12.5.2 Continuing calibration percent drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.5.3 Concentration in the extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration.

12.5.3.1 Average Response Factor

If the average of all the %RSDs of the response factors in the initial calibration is ≤ 15%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is}\overline{RF}}$$

12.5.3.2 Linear fit

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

Where: C_{ex} = Concentration in extract, µg/mL
R_x = Response for analyte
C_{is} = Concentration of internal standard
A = Intercept
B = Slope

12.5.3.3 Quadratic fit

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}{}^2}{R_{is}}\right)$$

Where: C = Curvature

12.5.4 The concentration in the sample is then calculated.

12.5.4.1 Aqueous Calculation

$$\text{Concentration, } \mu\text{g / L} = \frac{C_{ex} V_t}{V_o}$$

Where: V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 Ml extract will mean V_t = 10,000 µL. If half the base/neutral extract and half the acid extract are combined, V_t = 2,000.)

V_o = Volume of water extracted (mL)

12.5.5 Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis

$$\text{Concentration, } \mu\text{g / kg} = \frac{C_{ex} V_t}{W_s D}$$

Where: W_s = Weight of sample extracted or diluted in grams
D = (100 - % moisture in sample)/100, for a dry weight basis or one for a wet weight basis

12.6 MS/MSD percent recovery calculation.

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

Where: S_{SR} = Spike sample result
S_R = Sample result
S_A = Concentration equivalent of spike added

12.7 Relative % Difference calculation for the MS/MSD

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

Where: RPD = Relative percent difference
MS_R = Matrix spike result
MSD_R = Matrix spike duplicate result

12.8 Relative response factor calculation

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where: A_x = Area of the characteristic ion for the compound being measured
A_{is} = Area of the characteristic ion for the specific internal standard
C_x = Concentration of the compound being measured (µg/L)
C_{is} = Concentration of the specific internal standard (µg/L)

12.9 Calculation of TICs: The calculation of TICs) is identical to the above calculations with the following exceptions:

A_x = Area of the total ion chromatogram for the compound being measured

A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

12.10 Percent DDT breakdown

$$\% \text{ DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDarea}}$$

The total ion current areas are used for this calculation

12.11 Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002

13. METHOD PERFORMANCE

13.1 Method Detection Limit

13.1.1 Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in Policy CA-Q-S-006 and SOP NC-QA-021.

13.2 Initial Demonstration

13.2.1 Each laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of Laboratory Control Samples (LCS) containing all of the standard analytes for the method. For some tests, it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1.1 Four aliquots of the LCS are analyzed using the same procedures used to analyze samples, including sample preparation.

13.2.1.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.1.3 If any analyte does not meet the LCS acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3 Training Qualification

13.3.1 The Group/Team Leader has the responsibility to ensure this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.3.2 Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1 This section is not applicable to this procedure.

15. WASTE MANAGEMENT

15.1 All waste will be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2 Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by annual refresher training.

15.3 Waste Streams Produced by the Method

15.3.1 Vials containing sample extracts: These vials are placed in the vial waste located in the GC/MS laboratory.

16. REFERENCES

16.1 References

16.1.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III October 1994, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270C

16.1.2 J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry," Analytical Chemistry, 47, 995 (1975)

16.1.3 TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.4 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.5 Corporate Quality Management Plan (CQMP), current version

16.1.6 Revision History

Historical File:		Revision 2.1: 01/25/99		Revision 0: 05/28/08 (NC-MS-018)
(formerly CORP-MS-0001NC)		Revision 2.2: 03/27/00		Revision 1: 12/16/08
		Revision 2.3: 02/15/01		Revision 2: 10/26/10
		Revision 2.4: 05/29/01		
		Revision 2.5: 04/25/02		
		Revision 2.6: 08/15/02		
		Revision 2.7: 11/12/02		
		Revision 2.8: 01/23/03		
		Revision 2.9: 06/18/03		
		Revision 2.10: 02/24/04		
		Revision 2.11: 02/03/06		
		Revision 2.12: 03/01/07		

16.2 Associated SOPs and policies, current version

16.2.1 QA Policy, QA-003

16.2.2 Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.3 Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.4 Supplemental Practices for DoD Project Work, NC-QA-016

16.2.5 Standard and Reagents, NC-QA-017

16.2.6 Acceptable Manual Integration Practices, CA-Q-S-002

16.2.7 Calibration Curves (General), CA-Q-S-005

16.2.8 Section of Calibration Points, CA-T-P-002

17. MISCELLANEOUS

17.1 Modifications from Reference Method

17.1.1 A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2 The quantitation and qualifier ions from compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.2 Tables and Appendices

TABLE 1: TestAmerica Canton Standard Reporting Limits				
Analytes	**CAS Number**	**Water, µg/L**	**Soil, µg/kg**	**TCLP, mg/L**
1,1-Biphenyl	92-52-4	1	50	
1,2,4-Trichlorobenzene	120-82-1	1	50	
1,2-Dichlorobenzene	95-50-1	1	50	
1,3-Dichlorobenzene	541-73-1	1	50	
1,4-Dichlorobenzene	106-46-7	1	50	0.004
1-Methyl Naphthalene	90-12-0	0.2	6.67	
2,2'-oxybis(1-chloropropane)[1]	108-60-1	1	100	
2,4,5-Trichlorophenol	95-95-4	5	150	0.02
2,4,6-Trichlorophenol	88-06-2	5	150	0.02
2,4-Dichlorophenol	120-83-2	2	150	
2,4-Dimethylphenol	105-67-9	2	150	
2,4-Dinitrophenol	51-28-5	5	330	
2,4-Dinitrotoluene	121-14-2	5	200	0.02
2,6-Dinitrotoluene	606-20-2	5	200	
2-Chloronaphthalene	91-58-7	1	50	
2-Chlorophenol	95-57-8	1	50	
2-Methylnaphthalene	91-57-6	0.2	6.67	
2-Methylphenol	95-48-7	1	200	
2-Nitroaniline	88-74-4	2	200	
2-Nitrophenol	88-75-5	2	50	
3,3'-Dichlorobenzidine	91-94-1	5	100	
3-Nitroaniline	99-09-2	2	200	
3 & 4 Methylphenol	15831-10-4	2	400	0.04
4,6-Dinitro-2-methylphenol	534-52-1	5	150	
4-Bromophenyl phenyl ether	101-55-3	2	50	
4-Chloro-3-methylphenol	59-50-7	2	150	
4-Chloroaniline	106-47-8	2	150	
4-Chlorophenyl phenyl ether	7005-72-3	2	50	
4-Nitroaniline	100-01-6	2	200	
4-Nitrophenol	100-02-7	5	330	
Acenaphthene	83-32-9	0.2	6.67	
Acenaphthylene	208-96-8	0.2	6.67	
Aniline	62-53-3	5	330	
Anthracene	120-12-7	0.2	6.67	
Atrazine	1912-24-9	1	200	
Azobenzene	103-33-3	10	330	
Benzaldehyde	100-52-7	1	100	
Benzenethiol	108-98-5	10	330	
Benzidine	92-87-5	5	660	
Benzo(a)anthracene	56-55-3	0.2	6.67	
Benzo(a)pyrene	50-32-8	0.2	6.67	

TABLE 1: TestAmerica Canton Standard Reporting Limits				
Analytes	**CAS Number**	**Water, µg/L**	**Soil, µg/kg**	**TCLP, mg/L**
Benzo(b)fluoranthene	205-99-2	0.2	6.67	
Benzo(g,h,i)perylene	191-24-2	0.2	6.67	
Benzo(k)fluoranthene	207-08-9	0.2	6.67	
Benzoic acid	65-85-0	25	660	
Benzyl alcohol	100-51-6	5	330	
Bis(2-chloroethoxy)methane	111-91-1	1	100	
Bis(2-chloroethyl)ether	111-44-4	1	100	
Bis(2-ethylhexyl)phthalate	117-81-7	2	50	
Butyl benzyl phthalate	85-68-7	1	50	
Caprolactam	105-60-2	5	330	
Carbazole	86-74-8	1	50	
Chrysene	218-01-9	0.2	6.67	
Dibenz(a,h)anthracene	53-70-3	0.2	6.67	
Dibenzofuran	132-64-9	1	50	
Diethylphthalate	84-66-2	1	50	
Dimethyl phthalate	131-11-3	1	50	
Di-n-butyl phthalate	84-74-2	1	50	
Di-n-octylphthalate	117-84-0	1	50	
Fluoranthene	206-44-0	0.2	6.67	
Fluorene	86-73-7	0.2	6.67	
Hexachlorobenzene	118-74-1	0.2	6.67	0.02
Hexachlorobutadiene	87-68-3	1	50	0.02
Hexachlorocyclopentadiene	77-47-4	10	330	0.05
Hexachloroethane	67-72-1	1	50	
Indene	95-13-6	5	330	
Indeno(1,2,3-cd)pyrene	193-39-5	0.2	6.67	
Isophorone	78-59-1	1	50	
Naphthalene	91-20-3	0.2	6.67	
Nitrobenzene	98-95-3	1	100	0.004
N-nitrosodimethylamine	62-75-9	1	100	
N-Nitroso-di-n-propylamine	621-64-7	1	50	
N-Nitrosodiphenylamine	86-30-6	1	50	
Pentachlorophenol	87-86-5	5	150	0.04
Phenanthrene	85-01-8	0.2	6.67	
Phenol	108-95-2	1	50	
Pyrene	129-00-0	0.2	6.67	
Pyridine	110-86-1	1	100	0.02
Quinoline	91-22-5	5	330	
1,2,4,5-Tetrachlorobenzene	95-94-3	1	100	
1,3,5-Trinitrobenzene	99-35-4	5	1600	
1,3-Dinitrobenzene	99-65-0	2	330	
1,4-Naphthoquinone	130-15-4	50	330	
1-Naphthylamine	134-32-7	2	330	

TABLE 1: TestAmerica Canton Standard Reporting Limits				
Analytes	**CAS Number**	**Water, µg/L**	**Soil, µg/kg**	**TCLP, mg/L**
2,3,4,6-Tetrachlorophenol	58-90-2	10	100	
2,6-Dichlorophenol	87-65-0	5	150	
2-Acetylaminofluorene	53-96-3	10	330	
2-Naphthylamine	91-59-8	2	200	
2-Picoline	109-06-8	5	330	
2-secbutyl-4,6-dinitrophenol (Dinoseb2)	88-85-7	2	330	
3,3'-Dimethylbenzidine	119-93-7	5	330	
3-Methylcholanthrene	56-49-5	5	200	
4-Aminobiphenyl	92-67-1	5	330	
4-Nitroquinoline-1-oxide	56-57-5	5	330	
5-Nitro-o-toluidine	99-55-8	2	330	
7,12-Dimethylbenz(a)anthracene	57-97-6	2	330	
a,a-Dimethyl-phenethylamine	122-09-8	5	660	
Acetophenone	98-86-2	1	100	
Aramite	140-57-8	5	330	
Diallate[2]	2303-16-4	10	330	
Dibenz(a,j)acridine	224-42-0	5	330	
Dimethoate	60-51-5	2	330	
Disulfoton	298-04-4	2	330	
Ethyl methanesulfonate	62-50-0	2	330	
Famphur	52-85-7	10	3300	
Hexachloropropene	1888-71-7	5	0.02	
Isosafrole	120-58-1	5	330	
Methapyrilene	91-80-5	2	330	
Methyl methanesulfonate	66-27-3	2	330	
N-Nitrosodiethylamine	55-18-5	2	100	
n-Nitrosodi-n-butylamine	924-16-3	2	100	
N-Nitrosomethylethylamine	10595-95-6	2	100	
N-Nitrosomorpholine	59-89-2	2	330	
N-Nitrosopiperidine	100-75-4	2	330	
N-Nitrosopyrrolidine	930-55-2	2	50	
o,o,o-Triethyl-Phosphorothioate	126-68-1	2	330	
o-Toluidine	95-53-4	2	330	
p-(Dimethylamino)azobenzene	60-11-7	2	330	
p-Chlorobenzilate	510-15-6	2	330	
Pentachlorobenzene	608-93-5	2	100	
Pentachloroethane	76-01-7	20	330	
Pentachloronitrobenzene	82-68-8	2	330	
Phenacetin	62-44-2	2	330	
Phorate	298-02-2	2	330	
p-Phenylenediamine	106-50-3	40	660	
Pronamide	23950-58-5	2	330	

TABLE 1: TestAmerica Canton Standard Reporting Limits				
Analytes	**CAS Number**	**Water, µg/L**	**Soil, µg/kg**	**TCLP, mg/L**
Safrole	94-59-7	2	330	
Sulfotepp	3689-24-5	5	330	
Thionazin	297-97-2	2	330	
1,3-Dinitrobenzene	99-65-0	2	330	
1,4-Dinitrobenzene	100-25-4	2	100	
1,2-Dinitrobenzene	528-29-0	1	50	0.05
4,4'-Methylenebis(2-chloroaniline)	101-14-4	2		0.05
Diphenylamine	122-39-4	1	100	0.05
1,2-Diphenylhydrazine	122-66-7	1	50	0.05

1 *2,2'oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether.*

Skinner List Compound

Hexachlorophene is a required analyte for Appendix IX. This compound is not stable, and therefore not included in the calibration standard. The characteristic ions for hexachlorophene are searched for in the chromatogram (see Section 12.2.1).

Diphenylamine is a required compound for Appendix IX. N-nitrosodiphenylamine decomposes in the injection port to form diphenylamine. Therefore, these two compounds cannot be distinguished. Diphenylamine is not included in the calibration standard.

TABLE 1-A: TestAmerica Canton Standard Reporting Limits for LVI PAH				
Analytes	**CAS Number**	**Water, µg/L**	**Soil, µg/kg**	**TCLP, mg/L**
1,1-Biphenyl	95-52-4	1	50	0.01
1-Methylnaphthalene	90-12-120	0.2	6.67	0.05
2-Chloronaphthalene	91-58-7	1	50	0.05
2-Methylnaphthalene	91-57-6	0.2	6.67	0.05
Acenaphthylene	208-96-8	0.2	6.67	0.05
Acenaphthene	83-32-9	0.2	6.67	0.05
Anthracene	120-12-7	0.2	6.67	0.05
Benzo(a)anthracene	56-55-3	0.2	6.67	0.05
Benzo(b)fluoranthene	205-99-2	0.2	6.67	0.05
Benzo(k)fluoranthene	207-08-9	0.2	6.67	0.05
Benzo(a)pyrene	50-32-8	0.2	6.67	0.05
Benzo(g,h,I)perylene	191-24-2	0.2	6.67	0.05
Chrysene	218-01-9	0.2	6.67	0.05
Dibenzofuran	132-64-9	1	50	0.05
Dibenz(a,h)Anthracene	53-70-3	0.2	6.67	0.05
Fluoranthene	206-44-0	0.2	6.67	0.05
Fluorene	86-73-7	0.2	6.67	0.05
Hexachlorobenzene	118-74-1	0.2	6.67	0.02
Ideno(1,2,3-cd)pyrene	193-39-5	0.2	6.67	0.05
Phenanthrene	85-01-8	0.2	6.67	0.05

TABLE 2: TestAmerica Canton Michigan Program [1]			
Semivolatile	**CAS Number**	**Michigan Reporting Limits**	
		Aqueous µg/L	**Low Soil/Sediment µg/kg**
Acenaphthene	83-32-9	5	330
Acenaphthylene	208-96-8	5	330
Acetophenone	98-86-2	5	330
Anthracene	120-12-7	5	330
Atrzine	1912-24-9	5	330
Benzaldehyde	100-52-7	10	330
Benzo(a)anthracene	56-55-3	1	330
Benzo(a)pyrene	50-32-8	2	330
Benzo(b)fluoranthene	205-99-2	2	330
Benzo(g,h,i)perylene	191-24-2	5	330
Benzo(k)fluoranthene	207-08-9	5	330
1,1'-Biphenyl	92-52-4	10	330
4-Bromophenylphenyl ehter	101-55-3	5	330
Butylbenzylphthalate	85-68-7	5	330
di-n-Butylphthalate	84-74-2	5	330
Caprolactam	105-60-2	10	330
Carbazole	86-74-8	10	330
4-Chloroaniline	106-47-8	20	1700
bis(2-Chloroethoxy)methane	111-91-1	5	330
bis(2-Chloroethyl)ether	111-44-4	4	330
bis(2-Chloroisopropyl)ether	108-60-1	5	330
4-Chloro-3-Methylphenol	59-50-7	5	330
2-Chloronaphthalene	91-58-7	5	330
2-Chlorophenol	95-57-8	5	330
4-Chlorophenyl phenyl ether	7005-72-3	5	330
Chrysene	218-01-9	5	330
Dibenz(a,h)anthracene	53-70-3	2	330
Dibenzofuran	132-64-9	5	330
3,3'-Dichlorobenzidine	91-94-1	4	2000
2,4-Dichlorophenol	120-83-2	10	330
Diethylphthalate	84-66-2	5	330
2-4-Dimethylphenol	105-67-9	5	330
Dimethylphthalate	131-11-3	5	330

Semivolatile	CAS Number	Michigan Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
4,6-Dinitro-2-methylphenol	534-52-1	20	1700
2,4-Dinitrophenol	51-28-5	20	1700
2,4-Dinitrotoluene	121-14-2	5	330
2,6-Dinitrotoluene	606-20-2	5	330
bis(2-Ethylhexyl)phthalate	117-81-7	5	330
Fluoranthene	206-44-0	5	330
Fluorene	86-73-7	5	330
Hexachlorobenzene	118-74-1	5	330
Hexachlorobutadiene	87-68-3	5	330
Hexachlorocyclopentadiene	77-47-4	5	330
Hexachloroethane	67-72-1	5	330
Indeno(1,2,3-cd)pyrene	193-39-5	2	330
Isophorone	78-59-1	5	330
2-Methylnapthalene	91-57-6	5	330
2-Methylphenol	95-48-7	5	330
4-Methylphenol	106-44-5	5	330
Naphthalene	91-20-3	5	330
2-Nitroaniline	88-74-4	20	1700
3-Nitroaniline	99-09-2	20	1700
4-Nitroaniline	100-01-6	20	1700
Nitrobenzene	95-95-3	4	330
2-Nitrophenol	88-75-5	5	330
4-Nitrophenol	100-02-7	20	1700
N-Nitroso-di-n-propylamine	621-64-7	5	330
N-Nitrosodiphenylamine (diphenylamine)	62-75-9	5	330
di-n-Octylphthalate	117-84-0	5	330
Pentachlorophenol	87-86-5	20	800
Phenanthrene	85-01-8	5	330
Phenol	108-95-2	5	330
Pyrene	129-00-0	5	330
2,4,5-Trichlorophenol	95-95-4	5	330
2,4,6-Trichlorophenol	88-06-2	4	330

[1] *Reporting Limits are only for samples performed under the Michigan program.*

TABLE 3: Suggested Instrument Conditions	
Mass Range	35-500 amu
Scan Time	≤1 second/scan
Initial Column Temperature/Hold Time	60°C for 1 minutes, 50°C for 1 minute for LVI
Column Temperature Program	60 - 320°C at 35°C/min for 3 min 50 - 320°C at 35°C/min for 3 min for LVI
Final Column Temperature/Hold Time	320°C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature	250 - 300°C
Transfer Line Temperature	250 - 300°C
Source Temperature	According to manufacturer's Specifications
Injector	Grob-type, split / splitless
Sample Volume	0.5 µl, or 5.0 ul for LVI
Carrier Gas	Helium at 30 cm/sec

TABLE 4: DFTPP Key Ions and Ion Abundance Criteria	
Mass	**Ion Abundance Criteria**
51	30 – 80% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	25 - 75% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 – 9% of mass 198
275	10 – 30% of mass 198
365	> 0.75% of mass 198
441	Present, but less than mass 443
442	40 - 110% of mass 198
443	15 - 24% of mass 442

TABLE 5: Analytes in Approximate Retention Time Order and Characteristic Ions

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d5 (Surrogate Standard)	99	42	71
Benzaldehyde	77	105	106
Aniline	93	66	
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	79
2,2'-oxybis(1-chloropropane)[1]	45	77	79
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
Caprolactam	113	55	56
4-Chloro-3-methylphenol	107	144	142
1-Methylnaphthalene	142	141	115
2-Methylnaphthalene	142	141	115
Hexachlorocyclopentadiene	237	235	272
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
1,1'-Biphenyl	154	153	76

TABLE 5: Analytes in Approximate Retention Time Order and Characteristic Ions			
Analyte	**Primary**	**Secondary**	**Tertiary**
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153
2,6-Dinitrotoluene	165	63	89
Acenaphthene-d10 (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	84
4-Nitrophenol	109	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	182	77
N-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141
Azobenzene	77	182	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Atrazine	200	173	215
Pentachlorophenol	266	264	268
Phenanthrene-d10 (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125

TABLE 5: Analytes in Approximate Retention Time Order and Characteristic Ions			
Analyte	**Primary**	**Secondary**	**Tertiary**
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene -d12 (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277
2-Picoline	93	66	92
N-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	
3-Methylphenol	108	107	77
N-Nitrosopiperidine	114	42	55
o,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	75	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	
2-Naphthylamine	143	115	
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate	86	234	
Dimethoate	87	93	125

TABLE 5: Analytes in Approximate Retention Time Order and Characteristic Ions			
Analyte	**Primary**	**Secondary**	**Tertiary**
4-Aminobiphenyl	169		
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	160
Famphur	218	125	93
Methapyrilene	97	58	
Aramite 1	185	319	
Aramite 2	185	319	
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

TABLE 6: Method 8270C LCS Control Compounds	
LCS Compounds	**Spiking Level, Conc. Added = 20 ug/L**
1,2,4-Trichlorobenzene	20
Acenaphthene	20
2,4-Dinitrotoluene	20
Pyrene	20
N-Nitroso-di-n-propylamine	20
1,4-Dichlorobenzene	20
Pentachlorophenol	20
Phenol	20
2-Chlorophenol	20
4-Chloro-3-methylphenol	20
4-Nitrophenol	20
Acenaphthylene	20
Anthracene	20
Benzo(a)anthracene	20
Benzo(b)fluoranthene	20
Benzo(k)fluoranthene	20
Benzo(ghi)perylene	20
Benzo(a)pyrene	20
Bis(2-chloroehoxy)methane	20
Bis(2-chloroethyl)ether	20
Bis(2-chloroisopropyl)ether	20
Bis(2-ethylhexyl)phthalate	20
4-Bromophenyl phenyl ether	20
Butyl benzyl phthalate	20
Carbazole	20
2-Chloronaphthalene	20
Chrysene	20
Dibenz(ah)anthracene	20
Dibenzofuran	20
Di-n-butyl phthalate	20
1,2-Dichlorobenzene	20
1,3-Dichlorobenzene	20
1,4-Dichlorobenzene	20
Diethyl phthalate	20
2,4-Dimethylphenol	20
Dimethyl phthalate	20
2,4-Dinitrotoluene	20
2,6-Dinitrotoluene	20
Di-n-octyl phthalate	20
Fluoranthene	20

TABLE 6: Method 8270C LCS Control Compounds	
LCS Compounds	**Spiking Level, Conc. Added = 20 ug/L**
Fuorene	20
Hexachlorobenzene	20
Hexachlorobutadiene	20
Hexachloroethane	20
Indeno(1,2,3-cd)pyrene	20
Isophorone	20
2-Methylnaphthalene	20
2-Methylphenol	20
Naphthanlene	20
2-Nitroaniline	20
3-Nitroaniline	20
4-Nitroaniline	20
Nitrobenzene	20
2-Nitrophenol	20
4-Nitrophenol	20
N-Nitrosodimethylamine	20
N-Nitrosodiphenylamine	20
Phenanthrene	20
2,4,5-Trichlorophenol	20
2,4,6-Trichlorophenol	20

TABLE 7: Method 8270C All Analyte Spike Mix	
Acenaphthene	100
Acenaphthylene	100
Anthracene	100
Benzo(a)anthracene	100
Benzo(b)fluoranthene	100
Benzo(k)fluoranthene	100
Benzo(a)pyrene	100
Benzo(ghi)perylene	100
Benzyl butyl phthalate	100
Bis(2-chloroethyl)ether	100
Bis(2-chloroethoxy)methane	100
Bis(2-ethylhexyl)phthalate	100
Bis(2-chloroisopropyl)ether	100
4-Bromophenyl phenyl ether	100
2-Chloronaphthalene	100
4-Chlorophenyl phenyl ether	100
Chrysene	100
Dibenzo(a,h)anthracene	100
Di-n-butylphthalate	100
1,3-Dichlorobenzene	100
1,2-Dichlorobenzene	100
1,4-Dichlorobenzene	100
3,3'-Dichlorobenzidine	100
Diethyl phthalate	100
Dimethyl phthalate	100
2,4-Dinitrotoluene	100
2,6-Dinitrotoluene	100
Di-n-octylphthalate	100
Fluoranthene	100
Fluorene	100
Hexachlorobenzene	100
Hexachlorobutadiene	100
Hexachloroethane	100
Indeno(1,2,3-cd)pyrene	100

TABLE 7: Method 8270C All Analyte Spike Mix	
Isophorone	100
Naphthalene	100
N-Nitrosodi-n-propylamine	100
Phenanthrene	100
Pyrene	100
1,2,4-Trichlorobenzene	100
4-Chloro-3-methylphenol	100
2-Chlorophenol	100
2,4-Dichlorophenol	100
2,4-Dimethylphenol	100
2,4-Dinitrophenol	100
2-Methyl-4,6-dinitrophenol	100
2-Nitrophenol	100
4-Nitrophenol	100
Pentachlorophenol	100
Phenol	100
2,4,6-Trichlorophenol	100
Acetophenone	100
Atrazine	100
Caprolactam	100
Benzaldehyde	100
1,1′-Biphenyl	100
Benzoic Acid	100
1,4-Dioxane	100
Benzyl Alcohol	100
Carbazole	100
4-Chloroaniline	100
Dibenzofuran	100
Hexachlorocyclopentadiene	100
2-Methylnaphthalene	100
1-Methylnaphthalene	100
2-Methylphenol	100
4-Methylphenol	100
4-Nitroaniline	100
2-Nitroaniline	100
3-Nitroaniline	100

TABLE 7: Method 8270C All Analyte Spike Mix	
Pyridine	100
2,3,5,6-Tetrachlorophenol	100
2,4,5-Trichlorophenol	100
N-Nitrosodimethylamine	100
N-Nitrosodiphenylamine	100

TABLE 8: TCLP LCS Compounds	
LCS Compounds	**Spiking Level, mg/L in extract**
1,4-Dichlorobenzene	0.08
2,4-Dinitrotoluene	0.08
Hexachlorobenzene	0.08
Hexachlorobutadiene	0.08
Hexachloroethane	0.08
2-Methylphenol	0.08
3-Methylphenol	0.08
4-Methylphenol	0.08
Nitrobenzene	0.08
Pentachlorophenol	0.08
Pyridine	0.08
2,4,5-Trichlorophenol	0.08
2,4,6-Trichlorophenol	0.08

Recovery limits for the LCS and for matrix spikes are generated historical data, and are maintained by the QA Dept.

TABLE 9: Method 8270C Surrogate Compounds	
Surrogate Compounds	**Spiking Level, Conc. Added = 20 ug/L / 30 ug/L**
Nitrobenzene-d5	20
2-Fluorobiphenyl	20
Terphenyl-d14	20
Phenol-d5	30
2-Fluorophenol	30
2,4,6-Tribromophenol	30

Recovery limits for surrogates are generated from historical data, and are maintained by the QA department.

TABLE 10: Calibration Ranges		
Analyte	**Calibration Range**	**LVI Calibration Range**
Pyridine	0.5-25 ug/mL	0.05-2.5 ug/mL
N-nitrosodimethylamine	0.5-25 ug/mL	0.05-2.5 ug/mL
Aniline	0.5-25 ug/mL	0.05-2.5 ug/mL
Phenol	0.5-25 ug/mL	0.05-2.5 ug/mL
Bis(2-chloroethyl)ether	0.5-25 ug/mL	0.05-2.5 ug/mL
2-Chlorophenol	0.5-25 ug/mL	0.05-2.5 ug/mL
1,3-Dichlorobenzene	0.5-25 ug/mL	0.05-2.5 ug/mL
1,4-Dichlorobenzene	0.5-25 ug/mL	0.05-2.5 ug/mL
Benzyl alcohol	0.5-25 ug/mL	0.05-2.5 ug/mL
1,2-Dichlorobenzene	0.5-25 ug/mL	0.05-2.5 ug/mL
2-Methylphenol	0.5-25 ug/mL	0.05-2.5 ug/mL
2,2'-oxybis(1-chloropropane)[1]	0.5-25 ug/mL	0.05-2.5 ug/mL
4-Methylphenol	0.5-25 ug/mL	0.05-2.5 ug/mL
N-Nitroso-di-n-propylamine	0.5-25 ug/mL	0.05-2.5 ug/mL
Hexachloroethane	0.5-25 ug/mL	0.05-2.5 ug/mL
Nitrobenzene	0.5-25 ug/mL	0.05-2.5 ug/mL
Isophorone	0.5-25 ug/mL	0.05-2.5 ug/mL
2-Nitrophenol	0.5-25 ug/mL	0.05-2.5 ug/mL

TABLE 10: Calibration Ranges

Analyte	Calibration Range	LVI Calibration Range
2,4-Dimethylphenol	0.5-25 ug/mL	0.05-2.5 ug/mL
Benzoic acid	0.5-25 ug/mL	0.05-2.5 ug/mL
Bis(2-chloroethoxy)methane	0.5-25 ug/mL	0.05-2.5 ug/mL
2,4-Dichlorophenol	0.5-25 ug/mL	0.05-2.5 ug/mL
1,2,4-Trichlorobenzene	0.5-25 ug/mL	0.05-2.5 ug/mL
Naphthalene	0.1-25 ug/mL	0.01-2.5 ug/mL
4-Chloroaniline	0.5-25 ug/mL	0.05-2.5 ug/mL
Hexachlorobutadiene	0.5-25 ug/mL	0.05-2.5 ug/mL
4-Chloro-3-methylphenol	0.5-25 ug/mL	0.05-2.5 ug/mL
1-Methylnaphthalene	0.1-25 ug/mL	0.01-2.5 ug/mL
2-Methylnaphthalene	0.1-25 ug/mL	0.01-2.5 ug/mL
Hexachlorocyclopentadiene	0.5-25 ug/mL	0.05-2.5 ug/mL
2,4,6-Trichlorophenol	0.5-25 ug/mL	0.05-2.5 ug/mL
2,4,5-Trichlorophenol	0.5-25 ug/mL	0.05-2.5 ug/mL
2-Chloronaphthalene	0.1-25 ug/mL	0.01-2.5 ug/mL
2-Nitroaniline	0.5-25 ug/mL	0.05-2.5 ug/mL
Dimethyl phthalate	0.5-25 ug/mL	0.05-2.5 ug/mL
Acenaphthylene	0.1-25 ug/mL	0.01-2.5 ug/mL
3-Nitroaniline	0.5-25 ug/mL	0.05-2.5 ug/mL
Acenaphthene	0.1-25 ug/mL	0.01-2.5 ug/mL
2,4-Dinitrophenol	0.5-25 ug/mL	0.05-2.5 ug/mL
4-Nitrophenol	0.5-25 ug/mL	0.05-2.5 ug/mL
Dibenzofuran	0.1-25 ug/mL	0.01-2.5 ug/mL
2,4-Dinitrotoluene	0.5-25 ug/mL	0.05-2.5 ug/mL
2,6-Dinitrotoluene	0.5-25 ug/mL	0.05-2.5 ug/mL
Diethylphthalate	0.5-25 ug/mL	0.05-2.5 ug/mL
4-Chlorophenyl phenyl ether	0.5-25 ug/mL	0.05-2.5 ug/mL
Fluorene	0.1-25 ug/mL	0.01-2.5 ug/mL
4-Nitroaniline	0.5-25 ug/mL	0.05-2.5 ug/mL
46-Dinitro-2-methylphenol	0.5-25 ug/mL	0.05-2.5 ug/mL
N-Nitrosodiphenylamine	0.5-25 ug/mL	0.05-2.5 ug/mL
Azobenzene[2]	0.5-25 ug/mL	0.05-2.5 ug/mL
4-Bromophenyl phenyl ether	0.5-25 ug/mL	0.05-2.5 ug/mL
Hexachlorobenzene	0.5-25 ug/mL	0.05-2.5 ug/mL
Pentachlorophenol	0.5-25 ug/mL	0.05-2.5 ug/mL

TABLE 10: Calibration Ranges

Analyte	Calibration Range	LVI Calibration Range
Phenanthrene	0.1-25 ug/mL	0.01-2.5 ug/mL
Anthracene	0.1-25 ug/mL	0.01-2.5 ug/mL
Carbazole	0.5-25 ug/mL	0.05-2.5 ug/mL
Di-n-butyl phthalate	0.5-25 ug/mL	0.05-2.5 ug/mL
Fluoranthene	0.1-25 ug/mL	0.01-2.5 ug/mL
Benzidine	0.5-25 ug/mL	0.05-2.5 ug/mL
Pyrene	0.1-25 ug/mL	0.01-2.5 ug/mL
Butyl benzyl phthalate	0.5-25 ug/mL	0.05-2.5 ug/mL
3,3'-Dichlorobenzidine	0.5-25 ug/mL	0.05-2.5 ug/mL
Benzo(a)anthracene	0.1-25 ug/mL	0.01-2.5 ug/mL
Bis(2-ethylhexyl)phthalate	0.5-25 ug/mL	0.05-2.5 ug/mL
Chrysene	0.1-25 ug/mL	0.01-2.5 ug/mL
Di-n-octylphthalate	0.5-25 ug/mL	0.05-2.5 ug/mL
Benzo(b)fluoranthene	0.1-25 ug/mL	0.01-2.5 ug/mL
Benzo(k)fluoranthene	0.1-25 ug/mL	0.01-2.5 ug/mL
Benzo(a)pyrene	0.1-25 ug/mL	0.01-2.5 ug/mL
Indeno(1,2,3-cd)pyrene	0.1-25 ug/mL	0.01-2.5 ug/mL
Dibenz(a,h)anthracene	0.1-25 ug/mL	0.01-2.5 ug/mL
Benzo(g,h,i)perylene	0.1-25 ug/mL	0.01-2.5 ug/mL
Benzaldehyde	0.1-25 ug/mL	0.01-2.5 ug/mL
Caprolactam	0.5-25 ug/mL	0.05-2.5 ug/mL
1,1'-Biphenyl	0.1-25 ug/mL	0.01-2.5 ug/mL
Atrazine	0.5-25 ug/mL	0.05-2.5 ug/mL
2-Picoline	0.5-25 ug/mL	0.05-2.5 ug/mL
N-Nitrosomethylethylamine	0.5-25 ug/mL	0.05-2.5 ug/mL
Methyl methanesulfonate	0.5-25 ug/mL	0.05-2.5 ug/mL
N-Nitrosodiethylamine	0.5-25 ug/mL	0.05-2.5 ug/mL
Ethyl methanesulfonate	0.5-25 ug/mL	0.05-2.5 ug/mL
Pentachloroethane	0.5-25 ug/mL	0.05-2.5 ug/mL
Acetophenone	0.5-25 ug/mL	0.05-2.5 ug/mL
N-Nitrosopyrrolidine	0.5-25 ug/mL	0.05-2.5 ug/mL
N-Nitrosomorpholine	0.5-25 ug/mL	0.05-2.5 ug/mL
o-Toluidine	0.5-25 ug/mL	0.05-2.5 ug/mL
3-Methylphenol	0.5-25 ug/mL	0.05-2.5 ug/mL
N-Nitrosopiperidine	0.5-25 ug/mL	0.05-2.5 ug/mL

TABLE 10: Calibration Ranges

Analyte	Calibration Range	LVI Calibration Range
o,o,o-Triethyl-Phosphorothioate	0.5-25 ug/mL	0.05-2.5 ug/mL
a,a-Dimethyl-phenethylamine	0.5-25 ug/mL	0.05-2.5 ug/mL
2,6-Dichlorophenol	0.5-25 ug/mL	0.05-2.5 ug/mL
Hexachloropropene	0.5-25 ug/mL	0.05-2.5 ug/mL
p-Phenylenediamine	0.5-25 ug/mL	0.05-2.5 ug/mL
n-Nitrosodi-n-butylamine	0.5-25 ug/mL	0.05-2.5 ug/mL
Safrole	0.5-25 ug/mL	0.05-2.5 ug/mL
1,2,4,5-Tetrachlorobenzene	0.5-25 ug/mL	0.05-2.5 ug/mL
Isosafrole 1 + 2	0.5-25 ug/mL	0.05-2.5 ug/mL
1,4-Dinitrobenzene	0.5-25 ug/mL	0.05-2.5 ug/mL
1,4-Naphthoquinone	0.5-25 ug/mL	0.05-2.5 ug/mL
1,3-Dinitrobenzene	0.5-25 ug/mL	0.05-2.5 ug/mL
Pentachlorobenzene	0.5-25 ug/mL	0.05-2.5 ug/mL
1-Naphthylamine	0.5-25 ug/mL	0.05-2.5 ug/mL
2-Naphthylamine	0.5-25 ug/mL	0.05-2.5 ug/mL
2,3,4,6-Tetrachlorophenol	0.5-25 ug/mL	0.05-2.5 ug/mL
5-Nitro-o-toluidine	0.5-25 ug/mL	0.05-2.5 ug/mL
Thionazin	0.5-25 ug/mL	0.05-2.5 ug/mL
1,3,5-Trinitrobenzene	0.5-25 ug/mL	0.05-2.5 ug/mL
Sulfotepp	0.5-25 ug/mL	0.05-2.5 ug/mL
Phorate	0.5-25 ug/mL	0.05-2.5 ug/mL
Phenacetin	0.5-25 ug/mL	0.05-2.5 ug/mL
Diallate 1 + 2	0.5-25 ug/mL	0.05-2.5 ug/mL
Dimethoate	0.5-25 ug/mL	0.05-2.5 ug/mL
4-Aminobiphenyl	0.5-25 ug/mL	0.05-2.5 ug/mL
Pentachloronitrobenzene	0.5-25 ug/mL	0.05-2.5 ug/mL
Pronamide	0.5-25 ug/mL	0.05-2.5 ug/mL
Disulfoton	0.5-25 ug/mL	0.05-2.5 ug/mL
2-secbutyl-4,6-dinitrophenol (Dinoseb)	0.5-25 ug/mL	0.05-2.5 ug/mL
Methyl parathion	0.5-25 ug/mL	0.05-2.5 ug/mL
4-Nitroquinoline-1-oxide	0.5-25 ug/mL	0.05-2.5 ug/mL
Parathion	0.5-25 ug/mL	0.05-2.5 ug/mL
Isodrin	0.5-25 ug/mL	0.05-2.5 ug/mL

TABLE 10: Calibration Ranges		
Analyte	**Calibration Range**	**LVI Calibration Range**
Kepone	0.5-25 ug/mL	0.05-2.5 ug/mL
Famphur	0.5-25 ug/mL	0.05-2.5 ug/mL
Methapyrilene	0.5-25 ug/mL	0.05-2.5 ug/mL
Aramite 1 and 2	0.5-25 ug/mL	0.05-2.5 ug/mL
p-(Dimethylamino)azobenzene	0.5-25 ug/mL	0.05-2.5 ug/mL
p-Chlorobenzilate	0.5-25 ug/mL	0.05-2.5 ug/mL
3,3'-Dimethylbenzidine	0.5-25 ug/mL	0.05-2.5 ug/mL
2-Acetylaminofluorene	0.5-25 ug/mL	0.05-2.5 ug/mL
Dibenz (a,j)acridine	0.5-25 ug/mL	0.05-2.5 ug/mL
7,12-Dimethylbenz(a)anthracene	0.5-25 ug/mL	0.05-2.5 ug/mL
3-Methylcholanthrene	0.5-25 ug/mL	0.05-2.5 ug/mL

[1] *2,2'oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether.*

[2] *Azobenzene is formed by decomposition of 1,2-diphenyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.*

Note: *Nine calibrations standards are prepared varying in concentration from 0.01 ug/mL to 25 ug/mL. A minimum of 5 calibration concentrations will be used for initial calibration.*

SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Aniline	Acetophenone	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ethe
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,2-Dichlorobenzene	α,α-Dimethyl-	Dimethyl phthalate
Ethyl methanesulfonate	phenethylamine	2,4-Dinitrophenol
2-Fluorophenol (surr)	2,4-Dimethylphenol	2,4-Dinitrotoluene
Hexachloroethane	Hexachlorobutadiene	2,6-Dinitrotoluene
Methyl methanesulfonate	Isophorone	Fluorene
2-Methylphenol	2-Methylnaphthalene	2-Fluorobiphenyl (surr)
4-Methylphenol	Naphthalene	Hexachlorocyclopentadiene
N-Nitrosodimethylamine	Nitrobenzene	1-Naphthylamine
N-Nitroso-di-n-propylamine	Nitrobenzene-d_8 (surr)	2-Naphthylamine
Phenol	2-Nitrophenol	2-Nitroaniline
Phenol-d_6 (surr)	N-Nitrosodi-n-butylamine	3-Nitroaniline
2-Picoline	N-Nitrosopiperidine	4-Nitroaniline
	1,2,4-Trichlorobenzene	4-Nitrophenol
		Pentachlorobenzene
		1,2,4,5-Tetrachlorobenzene
		2,3,4,6-Tetrachlorophenol
		2,4,6-Tribromophenol (surr)
		2,4,6-Trichlorophenol
		2,4,5-Trichlorophenol

(surr) = surrogate

Table 11A: Method 8270Cand 8270D Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation		
1,4-Dichlorobenzene -d4	**Naphthalene -d8**	**Acenaphthene -d10**
Bis(2-chloroethyl)ether	Bis(2-chloroethoxy)methane	Acenaphthene
Bis(2-chloroisopropyl)ether	2,4-Dichlorophenol	Acenaphthylene
2-Chlorophenol	2,4-Dimethylphenol	2-Chloronaphthalene
1,2-Dichlorobenzene	Hexachlorobutadiene	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	Isophorone	Diethyl phthalate
1,4-Dichlorobenzene	Nitrobenzene	Dimethyl phthalate
2-Fluorophenol (surrogate)	Nitrobenzene-d_5 (surrogate)	2,4-Dinitrophenol
Hexachloroethane	2-Nitrophenol	2,4-Dinitrotoluene
N-Nitroso-di-n-propylamine	1,2,4-Trichlorobenzene	2,6-Dinitrotoluene
Phenol	Acetophenone	Fluorene
Phenol-d6 (surrogate)	Benzoic acid	2-Fluorobiphenyl (surrogate)
Aniline	4-Chloroaniline	4-Nitrophenol
Benzyl alcohol	4-Chloro-3-methylphenol	2,4,6-Tribromophenol(surrogate)
Ethyl methanesulfonate	2,6-Dichlorophenol	2,4,6-Trichlorophenol
Methyl methanesulfonate	a,a-Dimethylphenethylamine	
2-Methylphenol	2-Methylnaphthalene	Dibenzofuran
	Naphthalene	Hexachlorocyclopentadiene
N-Nitrosodimethylamine		1-Naphthylamine
2-Picoline	N-Nitrosodi-n-butylamine	2-Naphthylamine
	N-Nitrosopiperidine	2-Nitroaniline
		3-Nitroaniline
		4-Nitroaniline
		Pentachlorobenzene
		1,2,4,5-Tetrachlorobenzene
		2,3,4,6-Tetrachlorophenol
		2,4,5-Trichlorophenol

Table 11B: Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation		
Phenanthrene -d10	**Chrysene-d12**	**Perylene -d12**
Anthracene	Benzo(a)anthracene	Benzo(b)fluoranthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl)phthalate	Benzo(k)fluoranthene
Di-n-butyl phthalate	Chrysene	Benzo(g,h,i)perylene
4,6-Dinitro-2-methylphenol	3,3'-Dichlorobenzidine	Benzo(a)pyrene
Fluoranthene	Pyrene	Dibenz(a,h)anthracene
Hexachlorobenzene	Terphenyl-dl4 (surrogate)	Di-n-octylphthalate
Pentachlorophenol	Benzidine	Indeno(1,2,3-cd)pyrene
Phenanthrene	3,3'-Dichlorobenzidine	3-Methylcholanthrene
4-Aminobiphenyl	Butyl benzyl phthalate	Dibenz(a,j)acridine
Diphenylamine	p-Dimethyl aminoazobenzene	7,12-Dimethylbenz(a)anthracene
N-Nitrosodiphenylamine		
Pentachloronitrobenzene		
Phenacetin		
Pronamide		

Table 12: Recommended Minimum Response Factor Criteria for Initial and Continuing Calibration Verification	
Semivolatile Compounds	**Minimum Response Factor (RF)**
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Actophenone	0.010
4-Methylphenol	0.600
N-Nitros-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroanaline	0.010

Table 12: Recommended Minimum Response Factor Criteria for Initial and Continuing Calibration Verification	
Semivolatile Compounds	**Minimum Response Factor (RF)**
Hexachlorobutadiene	0.010
Caprolactuam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroanaline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Dithyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenylether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phananthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700

Table 12: Recommended Minimum Response Factor Criteria for Initial and Continuing Calibration Verification	
Semivolatile Compounds	**Minimum Response Factor (RF)**
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,I)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

Appendix A: Acronyms

CCC	Calibration Check Compounds
CCV	Continuing Calibration Verification
DFTPP	Decafluorotriphenylphosphine
DoD	Department of Defense
DQO	Data Quality Objectives
EH&S	Environmental Health and Safety
EICP	Extracted Ion Current Profile
GC/MS	Gas Chromatograph/Mass Spectrometer
ICV	Initial Calibration Verification
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
LLCCV	Low Level Continuing Calibration Verification
LVI	Large Volume Injection
MB	Method Blank
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheets
NCM	Non Conformance Memo
OSHA	Occupational Safety and Health Administration
OVAP	Ohio Voluntary Action Program
QAM	Quality Assurance Manual
PAH	Polycyclic Aromatic Hydrocarbons
PPM	Parts Per Million
PTFE	Polytetrafluoroethylene
QA/QC	Quality Assurance/Quality Control
RPD	Relative Percent Difference
RSD	Relative Standard Difference
RSE	Relative Standard Error
SPCC	System Performance Check Compounds
SOP	Standard Operational Procedure
STEL	Short Term Exposure Limit

TWA	Time Weighted Average

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TestAmerica Canton

SOP No. NC-GC-038, Rev. 3
Effective Date: 04/18/13

Title: GAS CHROMATOGRAPHIC ANALYSIS BASED ON METHODS 8000B, 8081A, 8081B, 8082, 8082A, 8151A, 8015B, and 8015C

Approvals (Signature/Date):

Technology Specialist	4/18/2013 Date	Health & Safety Coordinator	4/18/2013 Date
Quality Assurance Manager	4/18/2013 Date	Laboratory Director	4/18/2013 Date

This SOP was previously identified as SOP NC-GC-038, Rev 2, Dated 05/01/11

Facility Distribution No. _____ **Distributed To:_____**

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1. SCOPE AND APPLICATION

1.1. This SOP describes procedures for analysis of organic analytes by Gas Chromatography (GC). The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA). The main body of this SOP is based on SW846 Method 8000B. Individual analytes and methods are described in the appendices. See the list of appendices noted in the Table of Contents to determine the appropriate section of the SOP. Reporting limits are listed in each appendix.

2. SUMMARY OF METHOD

2.1. In general, semivolatile analytes in aqueous samples are prepared for analysis using continuous or separatory funnel liquid / liquid extraction (SOP NC-OP-037 and NC-OP-038). Solid samples are prepared using sonication or soxhlet (SOP NC-OP-039 and NC-OP-040). After the initial preparation step, the sample is introduced to the GC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. Internal or external standardization procedures are used as specified in the method appendices.

3. DEFINITIONS

3.1. Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms and acronyms used in this document.

4. INTERFERENCES

4.1. Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results. In particular, this is a problem with non-selective detectors such as the Flame Ionization Detector (FID). See Appendices A through D for interferences specific to individual tests and suggested corrective actions. All glassware is cleaned per SOP NC-QA-014.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that prevents splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Refer to the TestAmerica Canton Corporate Environmental Health and Safety Manual for a complete description of personal protection equipment. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated must be removed and discarded; other gloves must be cleaned immediately. Latex, Nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. It is recommended that analysts break up work tasks to avoid repetitive motion tasks, such as opening a large number of vials or containers in one time period.

5.5. Exposure to chemicals must be maintained as low as reasonably achievable. All samples with stickers that read "Caution/Use Hood!" must be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. Opened containers of neat standards must be handled in a fume hood.

5.7. Sample extracts and standards, which are in a flammable solvent, must be stored in an explosion-proof refrigerator.

5.8. When using hydrogen gas as a carrier, all precautions listed in the CSM must be observed.

5.9. Standard preparation and dilution must be performed inside an operating fume hood.

5.10. The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.11. There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.12. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. An analytical system complete with a gas chromatograph is required. A data system capable of measuring peak area and/or height is required. Recommended equipment and supplies for individual methods are listed in each method appendix.

7. REAGENTS AND STANDARDS

7.1. Stock Standards

7.1.1. Stock standards are purchased as certified solutions or prepared from pure solutions. Other stock standard solutions are stored as recommended by the manufacturer. All stock standards must be protected from light. Stock standard solutions must be brought to room temperature before using.

7.1.2. Semivolatile stock standard solutions must be replaced after one year. Stock standards of gases must be replaced at least every week, unless the acceptability of the standard is demonstrated (less than 20% drift from the initial calibration is an acceptable demonstration). Other volatile stock standards must be replaced every six months or sooner if comparison with check standards prepared from an independent source indicates a problem.

7.1.3. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampoule is opened, if the standard is supplied in a sealed ampoule. If vendor-supplied standard has an earlier expiration date then that date is used. Refer to SOP NC-QA-017, Standards and Reagents, for additional information. The standard preparation information is detailed in the LIMS standards and reagents module.

7.2. Calibration Standards

7.2.1. Semivolatile Calibration Standards

7.2.1.1. Semivolatile calibration standards are prepared as dilutions of the stock standards. Surrogates and internal standards are used as specified in the method appendices. Semivolatile calibration solutions must be refrigerated at $\leq$6°C and protected from light. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

7.3. Gases for carrier and make-up: Hydrogen, Nitrogen, Zero Air.

7.4. Quality control (QC) Standards

8. SAMPLE PRESERVATIONAND STORAGE

8.1. The holding time for semivolatile extracts is 40 days from extraction to analysis. Samples must be refrigerated at $\leq$6°C.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.2. For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2. Batch Definition

9.2.1. Batches are defined at the sample preparation stage. Batches must be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the TestAmerica North Canton QC Program document (Policy QA-003) for further details of the batch definition. Ohio VAP projects must reference this SOP instead of policy QA-003 for information on QC.

9.2.2. Quality Control Batch

9.2.2.1. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Laboratory generated QC samples (Method Blank, Laboratory Control Sample (LCS), matrix spike / spike duplicate (MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the matrix spike / spike duplicate (MS/MSD) may be replaced with a matrix spike and sample duplicate (MS/SD).

9.3. Control Limits

9.3.1. In-house historical control limits may be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined periodically. The recovery limits are mean recovery ± 3 standard deviations, unless that limit is tighter than the calibration criteria, in which case limits may be widened. Project or program specific control limits may be used in place of in-house limits. Refer to Policy QA-003 for more details.

9.3.2. These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries must be reported unless the dilution is more than 5X.

9.3.3. All surrogate, Laboratory Control Sample (LCS), and Matrix Spike (MS) recoveries (except for dilutions) must be entered into LIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes must be reported for all dilutions.

9.3.4. Refer to the QC Program document (Policy QA-003) for further details of control limits.

9.4. Surrogates

9.4.1. All methods must use surrogates to the extent possible. Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits. Surrogate recoveries must be met in the method blank (MB) and Laboratory Check Samples (LCS). If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure instrument performance is acceptable.
- Recalculate the data and/or re-analyze the extract if either of the above checks reveals a problem.

- The decision to re-analyze or flag the data must be made in consultation with the client. It is only necessary to reprepare / re-analyze a sample once to demonstrate poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out-of-control results are not due to matrix effect.

  Note: For DoD QSM, all surrogates must meet criteria. For Ohio VAP Projects, all surrogates must meet criteria unless the samples are ND and the surrogates are out high. Reanalysis or repreparation of the samples is required if these criteria are not met.

9.4.2. If dual column analysis is used, the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in-control result is reported.

9.4.3. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate (MS/MSD), then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the matrix spike (MS) and matrix spike duplicate (MSD) are in control, then repreparation or flagging of the data is required. Repreparation includes the parent sample and matrix spike / spike duplicate (MS/MSD).

9.4.4. Refer to TestAmerica North Canton QC Program document (Policy QA-003) for further details of the corrective actions.

9.5. Method Blanks

9.5.1. For each batch of samples, analyze a method blank. The method blank consists of reagent water for aqueous semivolatile samples and sodium sulfate for semivolatile soils tests (Refer to SOPs NC-OP-037, NC-OP-038, NC-OP-039, and NC-OP-040 for details). For low-level volatile soils, the method blank consists of reagent water and Ottawa sand. For medium-level volatile solids, the method blank consists of methanol and Ottawa sand. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the method blank and affected samples will normally be required. Consultation with the client must take place. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted if sufficient volume of sample remains.

9.5.2. The method blank must not contain any analyte of interest at, or above, the reporting limit (except common laboratory contaminants, see below) or at, or above, 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

9.5.3. Re-extraction and re-analysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

9.5.4. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action must be taken in consultation with the client.

Note: For Ohio VAP projects, the result must be below the reporting limit or samples must be re-extracted unless the samples are non-detect.

9.5.5. Refer to TestAmerica North Canton QC Program document (Policy QA-003) for further details of the corrective actions.

9.5.6. Refer to SOP NC-QA-016 for further details concerning DoD Project Work.

9.6. Laboratory Control Samples (LCS)

9.6.1. For each batch of samples, analyze a Laboratory Control Sample (LCS). The Laboratory Control Sample (LCS) contains a representative subset of the analytes of interest, and must contain the same analytes as the matrix spike. The Laboratory Control Sample (LCS) may also contain the full set of analytes with a subset of control analytes. If any control analyte is outside the laboratory established historical control limits, corrective action must occur. All non-controlling compounds must attain a recovery of 10% or greater if the compound is on the client's list. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted if sufficient volume of sample remains.

9.6.2. If the batch is not re-extracted and re-analyzed, the reasons for accepting the batch must be clearly presented in the project record and the report.

9.6.3. If re-extraction and re-analysis of the batch is not possible due to limited sample volume or other constraints, the Laboratory Control Sample (LCS) is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6.4. The Laboratory Control Sample (LCS) must have acceptable surrogate recoveries. If surrogate recoveries are low, re-extraction of the Laboratory Control Sample (LCS) and affected samples will normally be required. Consultation with the client should take place. For Ohio VAP samples, all analytes must meet criteria or the samples must be re-extracted if sufficient volume of sample remains.

9.6.5. Refer to TestAmerica North Canton QC Program document (Policy QA-003) for further details of the corrective action.

9.6.6. If dual column analysis is used, the choice of which result to report is made in the same way as for samples (Section 12.1.2), unless one column is out of control, in which case the in control result is reported.

9.6.7. Laboratory Control Sample (LCS) compound lists are included in the appendices.

9.7. Matrix Spikes/Spike Duplicates (MS/MSD)

9.7.1. For each QC batch, analyze a matrix spike and matrix spike duplicate (MS/MSD). Spiking compounds and levels are given in the appendices. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory-specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur unless samples for this compound are ND. The initial corrective action must be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the Laboratory Control Sample (LCS) is within limits, then the laboratory operation is in control and analysis may proceed.
- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate (MS/MSD) and the Laboratory Control Sample (LCS), the laboratory is out of control and corrective action must be taken. Corrective action must normally include repreparation and re-analysis of the batch.
- If a matrix spike / matrix spike duplicate (MS/MSD) is not possible due to limited sample, then a Laboratory Control Sample (LCS) duplicate may be analyzed if required by client or regulatory programs. The recovery for each spike of the pair must be within established control limits. If the RPD is out of control, but both accuracy recoveries are within acceptance criteria, prepare an NCM and qualify report.
- The matrix spike / matrix spike duplicate (MS/MSD) must be analyzed at the same dilution as the unspiked sample, unless the matrix spike components would then be above the calibration range.

9.7.2. If dual column analysis is used, the choice of which result to report is made in the same way as for samples (Section 12.1.2), unless one column is out of control, in which case the in control result is reported.

9.8. Control Limits

9.8.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.8.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMs.

10. CALIBRATION AND STANDARDIZATION

10.1. Internal or external calibration may be used. In either event, prepare standards containing each analyte of interest at a minimum of five concentration levels. The low-level standard must be at, or below, the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in the appendices.

10.2. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, changing PID lamps or replacing the ECD detector. A new calibration is not required after clipping the column, replacing the septum or syringe, or other minor maintenance.

10.3. With the exception of Section 10.4 below, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve, AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least five points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points. Third order calibrations require at least seven points.

10.4. A level may be removed from the calibration if the reason can be clearly documented (for example, a broken vial or no purge run). A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be re-analyzed and the re-analysis used for the calibration. All initial calibration points in a single calibration curve must be analyzed without any changes to instrument conditions, and all points in a single calibration curve must be analyzed within 24 hours.

10.5. External Standard Calibration

10.5.1. Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach, introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected may be used to prepare a calibration curve.

$$\textit{Calibration Factor or Response Factor} \quad (CF) \text{ or } (RF)) = \frac{\textit{Area or Height of Peak}}{\textit{Mass Injected (ng)}}$$

Some data systems may use the inverse of this formula. This is acceptable so long as the same formula is used for standards and samples. It is also possible to use the concentration of the standard rather than the mass injected. (This would require changes in the equations used to calculate the sample concentrations). Use of peak area or height must be consistent. However, if matrix interferences would make quantitation using peak area inaccurate for a particular sample, then peak height may be used as a substitute.

10.6. Calibration Curve Fits

10.6.1. Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor may be used if the average % RSD of the response factors or calibration factors of all the analytes in the calibration standard taken together is ≤ 20%. The average % RSD is calculated by summing the RSD value for each analyte and dividing by the total number of analytes. NOTE: This is not allowed for Ohio VAP projects or Update IV Methods.

10.6.2. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits must only be used if the analyst has reason to believe that the average RF model does not fit the normal concentration/response behavior of the detector. Linear or quadratic curve fits may be used if the compounds have historically exhibited a non-linear response and cannot be used to extend the calibration range for compounds that normally exhibit a linear response, but within a narrower calibration range.

10.6.3. Average Response Factor

The average response factor may be used if the average percent relative standard deviation (% RSD) of all the response factors taken together is ≤ 20%.

The equation for average response factor is:

$$Average\ response\ factor = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where: n = Number of calibration levels

$\sum_{i=1}^{n} RF_i$ = Sum of response factors for each calibration level

10.6.4. Linear Regression

The linear fit uses the following functions:

10.6.4.1.External Standard

$$y = ax + b$$

or

$$x = \frac{(y - b)}{a}$$

Where: y = Instrument response
x = Concentration
a = Slope
b = Intercept

10.6.4.2.Internal Standard

$$C_s = \frac{\left(\frac{A_s C_{is}}{A_{is}}\right) - b}{a}$$

Where: C_s = Concentration in the sample
A_s = Area of target peak in the sample
A_{is} = Area of internal standard in the sample
C_{is} = Concentration of the internal standard

10.6.5. Quadratic Curve

The quadratic curve uses the following functions:

10.6.5.1.External standard

$$y = ax + cx^2 + b$$

Where c is the curvature

10.7. Evaluation of Calibration Curves

10.7.1. The percent relative standard error (% RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation.

10.7.2. The least squares regression line is calculated and used to calculate the predicted concentration for each level. The percent relative standard error is calculated as follows.

$$\%\ RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left(\frac{C_i - PC_i}{C_i}\right)^2}{(N-P)}}$$

Where:
N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
C_I = True concentration for level i
PC_i = Predicted concentration for level i

Note: When average response factors are used, % RSE is equivalent to % RSD.

10.8. The following requirements must be met for any calibration to be used.

- Response must increase with increasing concentration.
- If a curve is used, the calculated intercept of the curve at zero response must be less than ± the reporting limit for the analyte.
- The curve used must be < The average Relative Standard Error (RSD for average response factors) of the calibration points from _ 20%.

- Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

  Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (r) and Coefficient of Determination (r^2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on r. As a result, a curve may have a very good correlation coefficient (>0.995) while also having > 100% error at the low point.

10.9. Weighting of Data Points

10.9.1. In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. $1/Concentration^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and must be used if the data system has this capability.

10.10. Non-standard analytes are sometimes requested. For these analytes, it may be acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five-point initial calibration. This action must be with client approval. If the analyte is detected in any of the samples, a five-point initial calibration must be generated, and the sample(s) re-analyzed for quantitation.

10.11. Calibration Verification

10.11.1. 12-hour Calibration

10.11.1.1. The working calibration curve or RF must be verified by the analysis of a midpoint calibration standard at the beginning, after every 12 hours, and at the end of the analysis sequence.

10.11.2. Daily Calibration Verification

10.11.2.1. It may be appropriate to analyze a mid-point standard more frequently than every 12 hours. If these calibration verification standards are analyzed, requirements are the same as the 12-hour calibration with the exception that retention times are not updated.

10.11.2.2. Any individual compounds with % D < 15% meet the calibration criteria. The calibration verification is also acceptable if the average of the % D for all the analytes is < 15%, or as noted in individual test sections. This average is calculated by summing the entire absolute % D results in the calibration (including surrogates) and dividing by the number of analytes. Only ND or results below the RL are reported. Any analyte that is reportable as found must have a % difference of < 15% in the calibration verification or 12-hour calibration on the column used for quantitation. Refer to Section 12.1.2 for which result to report. Update IV does not allow use of grand mean. CCV must pass by ± 20%.

10.11.3. An ICV is analyzed immediately after an initial calibration. The acceptance criteria is ± 20%. If this is not met, a new initial calibration curve is analyzed.

10.11.4. It is not necessary to run a calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.11.5. Samples quantitated by external standard methods must be bracketed by calibration verification standards that meet the criteria listed above. The bracketing standards on the column used for calibration must meet the same criteria as the opening standards. Bracketing is not necessary for internal standard methods.

10.11.6. If the analyst notes that a CCV has failed and can document the reason for failure (e.g., no purge, broken vial, carryover from the previous sample etc.), then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria, then the preceding samples have been successfully bracketed. If adjustments to the instrument are performed before the repeat CCV, then the proceeding samples have not been successfully bracketed; but analysis may continue.

10.11.7. In general, it is not advisable to analyze repeat CCVs on unattended runs. If repeat CCVs are analyzed, then the first must serve as the bracketing standard for the preceding samples and the last must serve as the CCV for the following samples.

10.11.8. If highly contaminated samples are expected, it is acceptable to analyze solvent blanks or primers at any point in the run.

10.11.9. Percent Difference Calculation

10.11.9.1. Percent difference for internal and external methods is calculated as follows:

External standard

$$\%D = \frac{RF_c - \overline{RF}}{\overline{RF}} \times 100 \qquad \%D = \frac{CF_c - \overline{CF}}{\overline{CF}} \times 100$$

Where: RF_c and CF_c are the response and calibration factors from the continuing calibration

RF and CF are the average response & calibration factors from the initial calibration

10.11.10. Percent Drift Calculation

10.11.10.1. Percent drift is used for comparing the continuing calibration to a linear or quadratic curve. The criteria for percent drift are the same as for percent difference

$$\%\ Drift = \frac{Calculated\ Conc. - Theoretical\ Conc.}{Theoretical\ Conc.} \times 100\%$$

10.11.10.2. Corrective Actions for Continuing Calibration

10.11.10.3. If the overall average percent drift of all analytes is greater than ± 15%, corrective action must be taken. This may include clipping the column, changing the liner, or other minor instrument adjustments, followed by re-analyzing the standard. If the overall average percent drift still varies by more than ± 15%, a new calibration curve must be prepared.

10.11.10.4. Corrective Action for Samples

10.11.10.4.1. For external standard methods, any samples injected after the last good continuing calibration standard must be re-injected.

10.11.10.4.2. If the average percent drift for all the analytes in the calibration is over 15%; but all of the analytes requested for a particular sample have percent drift $\leq$15%, then the analysis is acceptable for that sample.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo. The Nonconformance Memo must be filed in the project file. Procedural deviations are not allowed for Ohio VAP Projects.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.3. Extraction

11.3.1. Extraction procedures are referenced in the SOPs NC-OP-037, NC-OP-038, NC-OP-039, and NC-OP-040, current revision.

11.4. Cleanup

11.4.1. Cleanup procedures are referenced in the SOP NC-OP-025, current revision.

11.5. Gas Chromatography

11.5.1. Chromatographic conditions for individual methods are presented in the appendices.

11.6. Sample Introduction

11.6.1. Semivolatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

11.7. Analytical Sequence

11.7.1. An analytical sequence starts with an initial calibration or a calibration verification. Refer to the individual method appendices (Appendices A, B, C, and D for method-specific details of calibration verifications and analytical sequences.

11.7.2. The calibration verification includes analysis of standards containing all single response analytes and updating the retention time windows.

11.7.3. If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a calibration verification.

11.8. Retention Time Windows

11.8.1. Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day over a three-day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used). For multi-response analytes

(e.g., Aroclors), use the retention times of major peaks. Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.

11.8.2. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with the mid-point of the initial calibration and each 12-hour calibration. The widths of the windows must remain the same until new windows are generated following the installation of a new column.

11.8.3. If the retention time window as calculated above is less than ± 0.05 minutes, use a retention time window appropriate for the analysis and run time. This allows for slight variations in retention times caused by sample matrix.

11.8.4. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

11.8.5. Retention time studies are filed in the laboratory.

11.8.6. Corrective Action for Retention Times

11.8.6.1. The retention times of all compounds in the 12-hour calibration or calibration verification standard must be within the retention time window. If this condition is not met, all samples analyzed after the last compliant standard must be re-analyzed, unless the following conditions are met for any compound that elutes outside the retention time window.

11.8.6.2. The retention time of that compound in the standard must be within a retention time range equal to twice the original window.

11.8.6.3. No peak that would be reportable must be present on the sample chromatogram within an elution time range equal to three times the original retention time window.

11.9. Daily Retention Time Windows

11.9.1. The center of the retention time windows determined in Section 11.8 is adjusted to the retention time of each analyte as determined in the 12-hour calibration standards or continuing calibration verification standards. (See Methods 8081A and 8082 Appendices B and C for exceptions for multi-response components.) The retention time windows must be updated at the beginning of each analytical sequence and with each 12-hour calibration or continuing calibration verification.

11.10. Procedural Variations

11.10.1. Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo and approved by a supervisor and QA/QC Manager. The Nonconformance Memo must be filed in the project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described. Procedural deviations are not allowed for Ohio VAP Projects.

12. DATAANALYSISAND CALCULATIONS

12.1. Qualitative Identification

12.1.1. Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if J flags are required. Normally confirmation is required on a second column; but if the detector is sufficiently specific or if the sample matrix is well enough defined, single column analysis may be adequate. In some cases, GC/MS confirmation may be required. Client-specific requirements may also define the need for second column confirmation and/or GC/MS confirmation. Refer to the appendices for test specific requirements for confirmation. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column at a concentration greater than the reporting limit (MDL if J flag confirmation required). Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system. Many programs require chromatograms before and after manual integration. Additional information on manual integration can be found in SOP CA-Q-S-002.

12.1.2. Dual column quantitation

For confirmed results, two approaches are available to the analyst:

a) the primary column approach, or
b) the better result approach

Both are acceptable to avoid the reporting of erroneous or unconfirmed data.

12.1.2.1. Primary column approach

12.1.2.2. The result from the primary column is normally reported. The result from the secondary column is reported if any of the following three bulleted possibilities are true.

- There is obvious chromatographic interference on the primary column
- The result on the primary column is 40% greater than the result on the secondary column
- Continuing or bracketing standard fails on the primary column but is acceptable on the secondary column. (If the primary column result is > 40% higher than the secondary and the primary column calibration fails, then the sample must be evaluated for re-analysis.)

12.1.2.3. Better result approach

The higher of the two results is normally reported. The higher result is considered better because the higher result is generally higher because of chromatographic interference. For Ohio VAP projects, the higher result must be reported unless the laboratory can demonstrate that a matrix interference caused the result to be elevated. For Ohio VAP projects both columns must meet calibration criteria. The lower result is reported if any of the following two bulleted possibilities are true.

- There is obvious chromatographic interference on the column with the higher result
- The continuing or bracketing calibration on the column with the higher result fails. (If the higher result is > 40% higher and the calibration on the column with the lower result fails, then the sample must be evaluated for re-analysis.)

12.1.3. If the Relative Percent Difference (RPD) between the responses on the two columns is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect and the results are qualified. RPD is calculated using the following formula:

$$RPD = \frac{|R - R_2|}{\frac{1}{2}(R_1 + R_2)}$$

Where: R=Result

12.1.4. Multi-response Analytes

12.1.4.1. For multi-response analytes, the analyst must use the retention time window, but must rely primarily on pattern recognition. The pattern of peaks will normally serve as confirmation.

12.1.5. The experience of the analyst must weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.2. Calibration Range

12.2.1. If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and re-analyzed. Dilutions must target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix.

12.3. Dilutions

12.3.1. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be re-analyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.3.1.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and only minor matrix peaks are detected, then the sample must be re-analyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination.

12.3.2. Reporting Dilutions

12.3.2.1. The most concentrated dilution with no target compounds above the calibration range must be reported. Other dilutions may be reported at client request if the lower dilutions will not cause detector saturation, column overload, or carryover. Analyst judgment and client site history will be factors in the reporting of dual dilutions.

12.4. Interferences

12.4.1. If peak detection is prevented by interferences, further cleanup must be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.5. Calculations

12.5.1. Capabilities of individual data systems may require the use of different formulas than those presented here. When this is the case, the calculations used must be shown to be equivalent and must be documented in an appendix attached to this document.

12.5.2. External Standard Calculations

12.5.2.1. Aqueous samples

$$Concentration\ (mg/L) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times V_s)}$$

Where:
A_x = Response for the analyte in the sample
V_i = Volume of extract injected, µL
D_f = Dilution factor
V_t = Volume of total extract, µL
V_s = Volume of sample extracted or purged, mL
CF = Calibration factor, area or height/ng

12.5.2.2. Non-aqueous Samples

$$\text{Concentration}(mg/kg) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times W)}$$

Where:
W = Weight of sample extracted or purged, g

12.5.3. Surrogate Recovery

12.5.3.1. Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation.

$$\%\ \text{Recovery} = \frac{Concentration\ (or\ amount)\ found}{Concentration\ (or\ amount)\ spiked} \times 100$$

12.5.4. Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002

13. METHOD PERFORMANCE

13.1. Method Detection Limit

13.1.1. Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOPs NC-QA-021 and CA-Q-S-006.

13.2. Initial Demonstration

13.2.1. Each laboratory must make a one-time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests, it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample must be equivalent to a mid-level calibration.

13.2.1.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in each appendix.

13.2.1.3. If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. Training Qualification

13.3.1. The Group/Team Leader has the responsibility to ensure an analyst who has been properly trained in its use and has the required experience performs this procedure.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste must be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method

15.2.1. The following waste streams are produced when this method is carried out.

15.2.1.1. Vials containing sample extracts. These vials are placed in the vial waste located in the GC/MS laboratory.

15.2.1.2. Tubes containing sample extracts for TPH, pesticides, PCBs, and herbicides. These capped tubes are placed in the PCB/flammable waste located the GC prep laboratory.

15.2.1.3. Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCB's must be segregated into their own waste stream. PCB wastes are collected in one of three waste streams, solid PCB, liquid PCB and PCB vial waste. PCB containing samples are located through a LIMS query and disposed of as PCB containing.

15.2.1.4. Extracted solid samples contaminated with methylene chloride/acetone or acetone/hexane. These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.1.5. Discarded samples. These samples are collected in the solid debris drum.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, and Section 8000B

16.2 TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.3 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.4 Corporate Quality Management Plan (CQMP), current version

16.5 Revision History

Historical File:		Revision 2.1: 08/12/96		Revision 0: 01/21/08 (NC-GC-038)
(formerly CORP-GC-0001NC)		Revision 3.0: 12/01/97		Revision 1: 01/15/09 (NC-GC-038)
		Revision 5.3: 11/18/99		Revision 2: 05/01/11 (NC-GC-038)
		Revision 5.4: 11/10/00		
		Revision 5.5: 03/16/01		
		Revision 5.6: 05/25/01		
		Revision 5.7: 10/01/03		
		Revision 5.8: 02/06/06		

16.2. Associated SOPs and Policies, current version

16.5.1 QA Policy, QA-003

16.5.2 Glassware Washing, NC-QA-014

16.5.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.5.4 Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.5.5 Standards and Reagents, NC-QA-017

16.5.6 Cleanup Procedures for Organic Extractable Samples, NC-OP-025

16.5.7 Supplemental Practices for DoD Project Work, NC-QA-016

16.5.8 Acceptable Manual Integration Practices, CA-Q-S-002

16.5.9 Calibration Curves (General), CA-Q-S-005

16.5.10 Section of Calibration Points, CA-T-P-002

16.5.11 Continuous Liquid / Liquid Extraction of Organic Compounds from Waters Based on Methods SW846 3520C and 600 Series and Waste Dilution Based on Method 3580A, NC-OP-037

16.5.12 Separatory Funnel Extraction of Organic Compounds from Waters Based on Methods SW846 3510C and 600 Series and Waste Dilution Based on Method, NC-OP-038

16.5.13 Sonication Extraction of Organic Compounds from Soils Based on Method SW846 3550C and Waste Dilution Based on Method 3580A, NC-OP-039

16.5.14 Soxhlet (Traditional) Extraction of Organic Compounds from Soils Based on Method SW846 3540C and Waste Dilution Based on Method 3580A, NC-OP-040

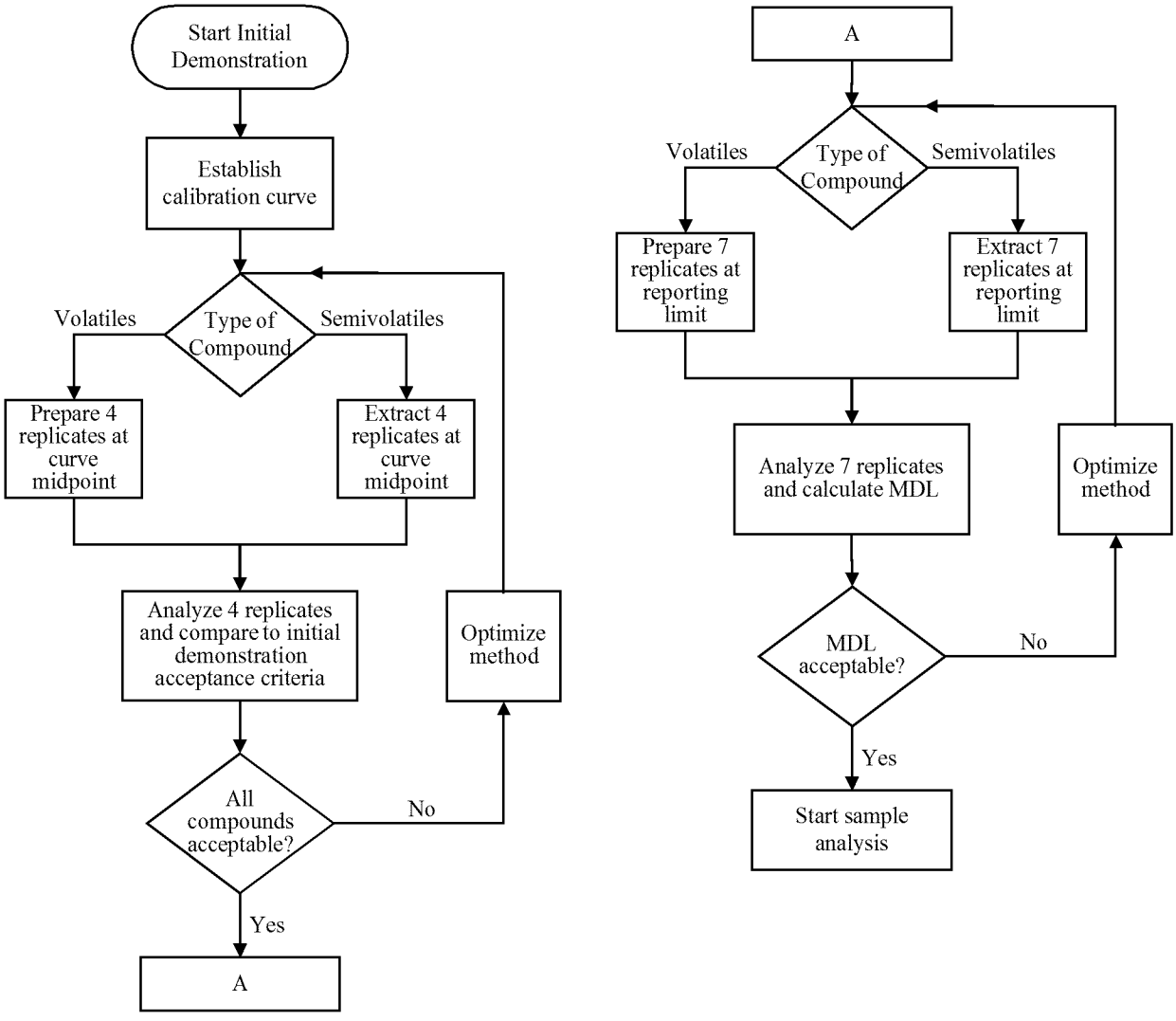
17. MISCELLANEOUS

17.1. Modifications from Reference Method

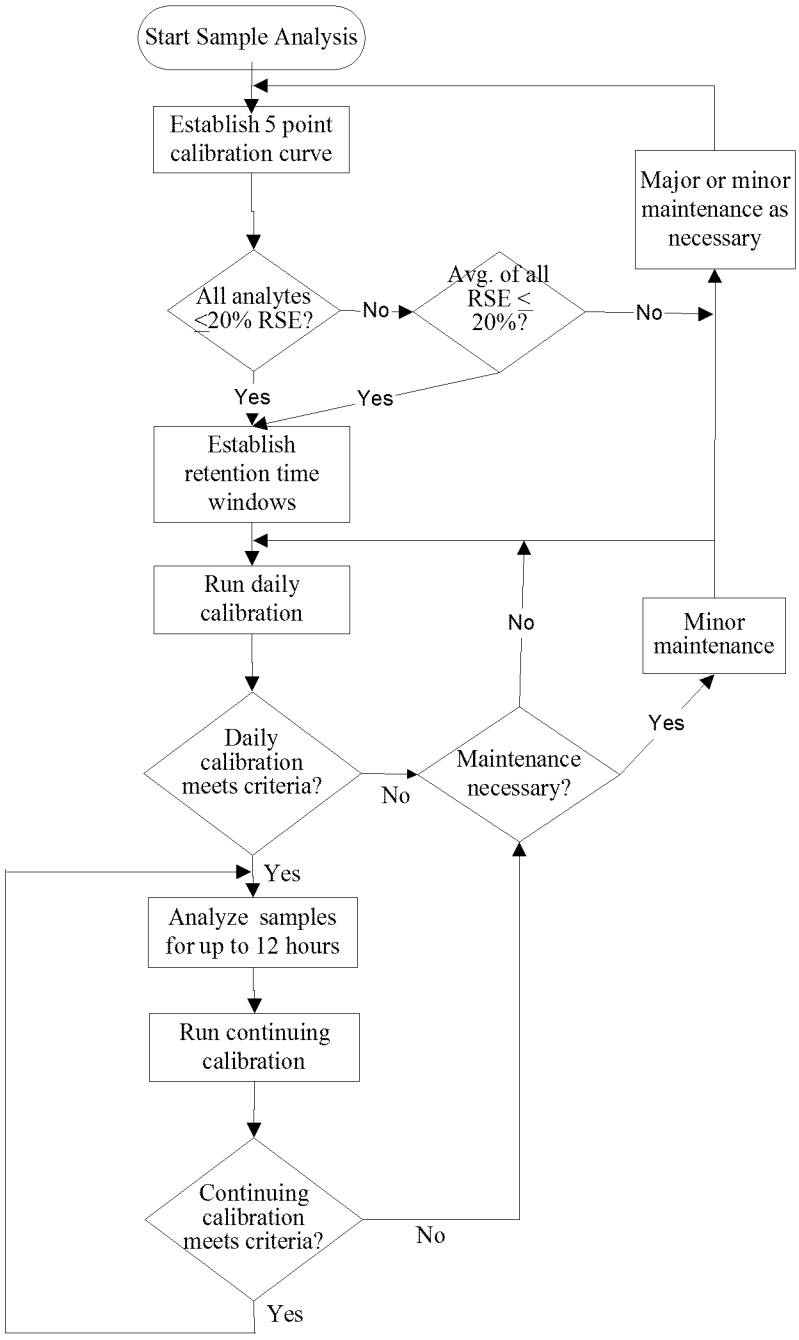
17.1.1. Chapter 1 of SW-846 states the method blank must not contain any analyte of interest at, or above, the Method Detection Limit. This SOP states the Method Blank must not contain any analyte of interest at, or above, the reporting limit. Common lab contaminants are allowed to be up to five times the reporting limit in the method blank following consultation with the client.

17.2. Flow Diagrams

17.2.1. Initial demonstration and MDL



17.2.2 Sample Analysis[1]



[1] This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.

1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8081A and 8081B is applied to the analysis of organochlorine pesticides by GC/ECD. This appendix is applicable to extracts derived from any matrix which are prepared according to the appropriate sample extraction SOPs.

1.2. Table B1 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

2. SUMMARY OF METHOD

2.1. This method presents conditions for the analysis of prepared extracts of organochlorine pesticides. The pesticides are injected onto the column and separated and detected by electron capture detection. Quantitation is by external standard methods.

3. DEFINITIONS

3.1. Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms and acronyms used in this document.

4. INTERFERENCES

4.1. Refer to the main body of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Avoiding contact with any plastic materials minimizes interferences from phthalates.

4.3. Sulfur will interfere and can be removed using procedures described in SOP NC-OP-025, Cleanup SOP.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Using hexane / acetone as the extraction solvent (rather than hexane / methylene chloride) will reduce the amount of interferences extracted.

5. SAFETY

5.1. Refer to main body of this SOP for general safety requirements.

5.2. The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, and the BHCs. Primary standards of these toxic compounds must be prepared in a hood.

5.3. All ^{63}Ni sources must be leak-tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.4. All ^{63}Ni sources must be inventoried every six months. If a detector is missing, the Director, EH&S must be immediately notified, and a letter sent to the NRC or local state agency.

6. EQUIPMENT AND SUPPLIES

6.1. A ^{63}Ni electron capture detector is required.

6.2. Refer to Table B2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

6.4 Autosampler vials, inserts, and caps

7. REAGENTS AND STANDARDS

7.1. Refer to the main body of this SOP for general requirements for reagents and supplies.

7.2. Refer to Table B3 for details of calibration standards. See the Standards Logbook for details on sample preparation.

7.3. Surrogate Standards

7.3.1. Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Refer to Tables B5 and B6 for details of surrogate standards.

7.4. Column Degradation Evaluation Mix

7.4.1. A mid-level standard containing 4,4'-DDT and Endrin and not containing any of their breakdown products must be prepared for evaluation of degradation of these compounds by the GC column and injection port. This solution also contains the surrogates. Refer to Table B4 for details of the column degradation evaluation mix.

8. SAMPLE COLLECTION, PRESERVATIONAND STORAGE

8.1. The holding time for pesticide extracts is 40 days from extraction to analysis. Samples must be refrigerated at $\leq$6°C.

9. QUALITY CONTROL

9.1. Refer to the main body of this SOP (Section 9) for general quality control procedures, including batch definition, requirements for method blanks, Laboratory Control Sample (LCS), matrix spike / spike duplicate (MS/MSD), surrogates, and control limits.

9.2. Refer to Table B5 for the components and levels of the Laboratory Control Sample (LCS) and matrix spikes / spike duplicates (MS/MSD) mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to the main body of this SOP for general calibration requirements.

10.2. Refer to Table B2 for recommended details of GC operating conditions. The conditions listed must result in resolution of all analytes listed in Table B1 on both columns.

10.3. Column Degradation Evaluation

10.3.1. Before any calibration runs, either initial or 12 hour, the column evaluation mix must be injected before each initial or daily calibration. The degradation of DDT and endrin must be

calculated (see Equations 9 and 10) and each shown to be less than 15% before calibration can proceed. This is only necessary if the target compound list includes DDT, Endrin, or any of their degradation products.

10.3.2. If the breakdown of DDT and/or endrin exceeds the limits given above, corrective action must be taken. This action may include:

10.3.3. Replacement of the injection port liner or the glass wool.

10.3.4. Cutting off a portion of the injection end of a capillary column.

10.3.5. Replacing the GC column.

10.4. Initial Calibration

10.4.1. Refer to Section 10 of the Method 8000B section of this SOP for details of calibration procedures. The low-level calibration standard must be at, or below, the reporting limit.

10.4.2. Refer to Table B7 for the initial calibration analytical sequence.

10.4.3. The response for each single-peak analyte must be calculated by the procedures described in the main body of this SOP.

10.4.4. The surrogate calibration curve is calculated from the AB mix. If there are resolution problems, then the A and B mixes may be analyzed separately.

10.4.5. For multi-component pesticides:

10.4.5.1. A calibration with a minimum of five points is used for multi-component pesticides (typically toxaphene and technical chlordane). Two options are possible; the same quantitation option must be used for standards and samples. Refer to Section 12.3 for guidance on which option to use.

10.4.5.2. A full calibration for any of the multi-component analytes is analyzed.

10.5. Daily 12-hour Calibration Verification

10.5.1. The 12-hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12-hour calibration. A mid-level calibration standard is used for the 12-hour calibration. For Method 8081B, the CCV acceptance criteria is ± 20%

10.5.2. At a minimum, the 12-hour calibration includes analysis of the breakdown mix followed by mid-level standards of the AB mix. At a minimum, multi-component analytes are analyzed at the beginning of a sequence.

10.5.3. The retention time windows for any analytes included in the 12-hour calibration are updated.

10.6. Initial Calibration Verification (ICV)

10.6.1. An initial calibration verification (ICV) standard, from a second source, is analyzed immediately following the initial calibration. Acceptance criteria is ± 20%.If this is not met, a new initial calibration curve is analyzed.

10.7. Daily Continuing Calibration

10.7.1. A mid-level AB calibration mix is analyzed as the continuing calibration standard. At a minimum, this is analyzed after every 20 samples, including matrix spike / spike duplicate (ms/msd), Laboratory Control Sample (LCS), and method blanks. If 12 hours elapse, analyze the 12-hour standard sequence instead. The continuing calibration standard need not include multi-component analytes. If instrument drift is expected due to sample matrix or other factors, it may be advisable to analyze the continuing calibration standard more frequently.

11. PROCEDURE

11.1. Refer to the main body of this SOP for general procedural requirements.

11.2. Suggested gas chromatographic conditions are given in Table B2.

11.3. Allow extracts to warm to ambient temperature before injection.

11.4. The suggested analytical sequence is given in Table B7.

12. DATAANALYSISAND CALCULATIONS

12.1. Refer to the main body of this SOP for identification and quantitation of single component analytes.

12.2. Identification of Multi-Component Analytes

12.2.1. Retention time windows are also used for identification of multi-component analytes, but the "fingerprint" produced by major peaks of those compounds in the standard is used in tandem with the retention times to identify the compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.3. Quantitation of Multi-Component Analytes

12.3.1. Use 3-10 major peaks (or total area for quantitation) as described in Section 10.4.4, initial calibration of multi-component analytes.

12.3.2. If there are no interfering peaks within the envelope of the multi-component analyte, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.

12.3.2.1. Multiple peak option

12.3.3. This option is particularly valuable if toxaphene is identified but interferences make quantitation based on total area difficult. Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks. Alternatively, find the response of each of the 3-10 peaks per multi-peak pesticide, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be coeluting

with contaminant peaks from the quantitation. (i.e., peaks which are significantly larger than would be expected from the rest of the pattern.)

12.3.4. Chlordane may be quantitated either using the multiple peak option (Section 12.3.1, Appendix B), total area option (Section 12.3. 2., Appendix B), or by quantitation of the major components, α-chlordane, γ-chlordane and heptachlor.

12.4. Second column confirmation multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence. For Ohio VAP and DoD projects, both columns must meet criteria for positive detections.

12.5. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

12.6. Calculation of Column Degradation/% Breakdown (%B)

Equation 9

$$DDT\ \%B = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100$$

Where:

A_{DDD}, A_{DDE}, and A_{DDT} = the response of the peaks for 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT in the column degradation evaluation mix.

Equation 10

$$Endrin\ \%B = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_{E}} \times 100$$

Where:

A_{EK}, A_{EA}, and A_E = the response of endrin ketone, endrin aldehyde, and endrin in the column degradation evaluation mix.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced in the main body of this SOP.

14. POLLUTION PREVENTION

14.1. Refer to Section 14 of the main body of this SOP.

15. WASTE MANAGEMENT

15.1. Refer to Section 15 of the main body of this SOP.

16. REFERENCES

16.1. SW846, Update III, December 1996, Method 8081A

16.2. SW846, Revision 2, February 2007, 8081B

17. MISCELLANEOUS

17.1. Modifications from Reference Method - None

17.2. Tables

TABLE B1

Standard Analyte List and Reporting Limits for Methods 8081A and 8081B

Compound	Reporting Limit, µg/L or µg/kg		
	Water	Soil	Waste
Aldrin	0.05	1.7	50
α-BHC	0.05	1.7	50
β-BHC	0.05	1.7	50
δ-BHC	0.05	1.7	50
γ-BHC (Lindane)	0.05	1.7	50
α-Chlordane	0.05	1.7	50
γ-Chlordane	0.05	1.7	50
Chlordane (technical)	0.5	17	500
4,4'-DDD	0.05	1.7	50
4,4'-DDE	0.05	1.7	50
4,4'-DDT	0.05	1.7	50
Dieldrin	0.05	1.7	50
Endosulfan I	0.05	1.7	50
Endosulfan II	0.05	1.7	50
Endosulfan Sulfate	0.05	1.7	50
Endrin	0.05	1.7	50
Endrin Aldehyde	0.05	1.7	50
Heptachlor	0.05	1.7	50
Heptachlor Epoxide	0.05	1.7	50
Methoxychlor	0.1	3.3	100
Toxaphene	2.0	67	2000
APPENDIX IX ADD-ONs			
Diallate	1.0	33	1000
Isodrin	0.1	3.3	100
Chlorobenzilate	0.25	16.7	500
Kepone[1]	1.0	33	1000
Hexachlorobenzene	0.05	33	1000

[1] Kepone is sometimes requested for analysis by method 8081A. However, kepone may produce peaks with broad tails that elute later than the standard by up to a minute (presumably due to hemi-acetal formation). As a result kepone analysis by 8081A is unreliable and not recommended. Analysis by method 8270C is a possible alternative.
Note: alpha chlordane, gamma chlordane, and endrin ketone are not required for some projects. The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.
Ground water	1000 mL	5 mL
Low-level soil	30g	10 mL
High-level soil / waste	1g	10 mL

TABLE B2
Recommended Conditions for Methods 8081A and 8081B

Parameter	Recommended Conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	120°C for 1 min, 8.5°C/min to 285°C, , 6 min hold
Column 1	Rtx-CLPesticides 30m x 0.53mm id, 0.5µm
Column 2	Rtx-CLPesticideII 30m,0.53mm id, 0.5um
Injection	1µL
Carrier gas	Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

TABLE B3
Calibration Levels ng/mL for Methods 8081A and 8081B

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[2]
Individual Mix AB[1]						
Aldrin	5	10	25	50	100	200
g-BHC (Lindane)	5	10	25	50	100	200
Heptachlor	5	10	25	50	100	200
Methoxychlor	5	10	25	50	100	200
Dieldrin	5	10	25	50	100	200
Endosulfan I	5	10	25	50	100	200
Endosulfan II	5	10	25	50	100	200
4,4'-DDT	5	10	25	50	100	200
Endrin Aldehyde	5	10	25	50	100	200
Endrin Ketone	5	10	25	50	100	200
β-BHC	5	10	25	50	100	200
δ-BHC	5	10	25	50	100	200
α-BHC	5	10	25	50	100	200
4,4'-DDD	5	10	25	50	100	200
4,4'-DDE	5	10	25	50	100	200
Endosulfan Sulfate	5	10	25	50	100	200
Endrin	5	10	25	50	100	200
α-Chlordane [3]	5	10	25	50	100	200
γ-Chlordane [3]	5	10	25	50	100	200
Multi-component Standards						
Chlordane (Technical)	20	50	100	200	500	
Toxaphene	200	500	1000[5]	2000	5000	
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl	5	10	25	50	100	200

[1] Standards may be split into an A and B mix if resolution of all compounds on both columns is not obtained.
[2] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.
[3] Compounds may be used in lieu of running a daily technical Chlordane standard for samples that are non-detect for technical Chlordane.

TABLE B4

Column Degradation Evaluation Mix ng/mL for Methods 8081A and 8081B

Component	Concentration
4,4'-DDT	25
Endrin	25
Tetrachloro-m-xylene (Surrogate)	20
Decachlorobiphenyl (Surrogate)	20

TABLE B5

Laboratory Control Sample (LCS)/Matrix Spike/Spike Duplicate (MS/MSD) and Surrogate Spike levels µg/L or µg/kg for Methods 8081A and 8081B

	Aqueous	Soil	Waste
gamma BHC (Lindane)	1	33.3	1000
Aldrin	1	33.3	1000
Heptachlor	1	33.3	1000
Dieldrin	1	33.3	1000
Endrin	1	33.3	1000
Alpha BHC	1	33.3	1000
Beta BHC	1	33.3	1000
Delta BHC	1	33.3	1000
Gamma BHC	1	33.3	1000
4,4'DDD	1	33.3	1000
4,4'DDE	1	33.3	1000
4,4'DDT	1	33.3	1000
Endosulfan I	1	33.3	1000
Endosulfan II	1	33.3	1000
Endosulfan Sulfate	1	33.3	1000
Heptachlor Expoxide	1	33.3	1000
Methoxychlor	1	33.3	1000
Endrin Ketone	1	33.3	1000
Endrin Aldehyde	1	33.3	1000
Alpha-chlordane	1	33.3	1000
Gamma-chlordane	1	33.3	1000
Tetrachloro-m-xylene (Surrogate)	0.20	6.7	200
Decachlorobiphenyl (Surrogate)	0.20	6.7	200

TABLE B6	
Laboratory Control Sample (LCS)/Matrix Spike/Spike Duplicate (MS/MSD) and Surrogate Spike levels for TCLP µg/L or µg/kg for Methods 8081A and 8081B	
	Aqueous
Heptachlor	2
Heptachlor epoxide	2
Lindane	2
Endrin	2
Methoxychlor	4

TABLE B7
Suggested Analytical Sequence for Methods 8081A and 8081B

Initial Calibration

Solvent blank (optional)	
Primer if needed	
Breakdown Mix	
Individual mix AB	All levels
ICV	
Technical Chlordane	Level 3[1]
Toxaphene	Level 3[1]
Up to 20 samples unless 12 hours comes first)	
Solvent blank (optional)	
Individual mix AB	Mid level (Continuing calibration)
Samples	
After 12 hours:	
Breakdown mix	
Individual mix AB	
Any other single component analytes	
Any multi-component analytes	

[1] A curve with a minimum of five points for any of the multi-component analytes may be included.

Note: A solvent blank or primer may be analyzed at any time during the sequence when highly contaminated samples are expected. A solvent blank or primer may not be analyzed as routine immediately prior to standards.

Note: The initial primer is used if the instrument has been idle for a period of time.

12 -Hour Calibration

At least every 12 hours, counting from the start of the initial calibration or from the start of the last daily calibration, the retention time windows must be updated using the Individual mix AB and a PEM must be analyzed if the analysis is to continue.

1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8082 or 8082A is applied to the analysis of polychlorinated biphenyls (PCB) by GC/ECD. This appendix is applicable to extracts derived from any matrix which are prepared according to the appropriate sample extraction SOPs. PCBs are determined and quantitated as Aroclor mixes.

1.2. Tables C1 and C5 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

Note: SW-846 method 8082 and 8082A provides incomplete guidance for determination of individual PCB congeners. This SOP does not include directions for congener specific analysis.

2. SUMMARY OF METHOD

2.1. This method presents conditions for the analysis of prepared extracts of PCBs. The PCBs are injected onto the column and separated and detected by electron capture detection. Quantitation is by external standard methods.

3. DEFINITIONS

3.1. Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms and acronyms used in this document.

4. INTERFERENCES

4.1. Refer to the main body of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Avoiding contact with any plastic materials minimizes interferences from phthalates.

4.3. Sulfur will interfere and can be removed using procedures described in SOP NC-OP-025.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts. These cleanup procedures are included in SOP NC-OP-025.

5. SAFETY

5.1. Refer to the main body of this SOP for general safety requirements.

5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

5.3. All ^{63}Ni sources must be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.4. All ^{63}Ni sources must be inventoried every six months. If a detector is missing, the EH&S Director must be immediately notified and a letter sent to the NRC or local state agency.

6. EQUIPMENT AND SUPPLIES

6.1. A ^{63}Ni electron capture detector is required.

6.2. Refer to Table C2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

6.4 Autosampler vials, inserts, and caps

7. REAGENTS AND STANDARDS

7.1. Refer to the main body of this SOP for general requirements for reagents and supplies.

7.2. Refer to Table C3 for details of calibration standards. See the Standards Logbook for details on sample preparation.

7.3. Surrogate Standards

7.3.1. Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Refer to Table C4 for details of surrogate standards.

8. SAMPLE COLLECTION, PRESERVATIONAND STORAGE

8.1. The holding time for PCB extracts is 40 days from extraction to analysis. Samples must be refrigerated at $\leq$6°C.

9. QUALITY CONTROL

9.1. Refer to main body of this SOP for general quality control procedures, including batch definition, requirements for method blanks, Laboratory Control Sample (LCS), matrix spikes / spike duplicate (MS/MSD), surrogates, and control limits.

9.2 Refer to Table C4 for the components and levels of the Laboratory Control Sample (LCS) and matrix spikes / spike duplicates (MS/MSD) mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to the main body of this SOP for general calibration requirements.

10.2 Update IV recommends analysis of DDT and analogs DDD and DDE prior to calibration to assure there is not interference with major 1254 peaks.

10.2. Initial Calibration

10.2.1. Refer to Table C6 for the initial calibration analytical sequence.

10.2.2. The response for each Aroclor must be calculated by the procedures described in the main body of this SOP with the following modifications.

10.2.3. A minimum five-point calibration of all Aroclors is generated. The average response factor is used to quantitate Aroclors. The low-level standard must be at or below the reporting limit. The other standards define the working range of the detector.

10.2.4. The high and low standards for the initial calibration of 1016 / 1260 define the acceptable quantitation range for the other Aroclors. If any Aroclor is determined above this concentration, the extract must be diluted and re-analyzed.

Note: For Ohio VAP, Aroclor 1268 may be analyzed. In order to meet project-specific reporting limits, a lower concentration standard may be added to the calibration curve.

10.2.5. If the analyst knows that a specific Aroclor is of interest for a particular project, that Aroclor may be used for the calibration rather than the 1016 / 1260 mix.

10.2.6. The surrogate calibration curve is calculated from the Aroclor 1016/1260 mix. Surrogates in the other calibration standards are used only as retention time markers.

10.2.7. The following is used for the quantitation of all Aroclors. The same quantitation option must be used for standards and samples.

10.2.7.1. Multiple peak option.

10.2.7.2. Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks.

10.3. Daily 12-Hour Continuing Calibration

10.3.1. The 12-hour calibration verification must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12-hour calibration. For Method 8082A, the CCV acceptance criterion is ± 20%.

10.3.2. At a minimum, the 12-hour calibration includes analysis of the Aroclor 1260 / 1016 mix.

10.3.3. Other Aroclors are included in the daily calibration check.

10.3.4. The retention time windows for any analytes included in the daily calibration and CCVs are updated.

10.3.5. For this method, samples must be bracketed with successful calibration verification runs.

10.4. Initial Calibration Verification (ICV)

10.4.1. An initial calibration verification (ICV) standard, from a second source, is analyzed immediately following the initial calibration. An acceptance criterion is ± 20%. If this is not met, a new initial calibration curve is analyzed.

10.5. Daily Calibration Verification Standards

10.5.1. The Aroclor 1260/1016 calibration mix is analyzed as the calibration verification standard. This is analyzed after every 20 samples, including matrix spikes, Laboratory Control Sample (LCS), and method blanks. (Depending on the type of samples, it may be advisable to analyze verifications more frequently in order to minimize reruns.)

10.5.2. A mid-level standard is used for the calibration verification.

11. PROCEDURE

11.1. Refer to the main body of this SOP for general procedural requirements.

11.2. Suggested gas chromatographic conditions are given in Table C2.

11.3. Allow extracts to warm to ambient temperature before injection.

11.4. The suggested analytical sequence is given in Table C6.

12. DATAANALYSISAND CALCULATIONS

12.1. Identification of Aroclors

12.1.1. Retention time windows are used for identification of Aroclors, but the "fingerprint" produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.1.2. A clearly identifiable Aroclor pattern serves as confirmation of single column GC analysis.

12.2. Quantitation of Aroclors

12.2.1. Use 3-10 major peaks or total area for quantitation

12.2.2. If the analyst believes that a combination of Aroclor 1254 and 1260 or a combination of 1242, 1248 and 1232 is present, then only the predominant Aroclor is quantitated and reported; but the suspicion of multiple Aroclors is discussed in the narrative. If well-separated Aroclor patterns are present, then multiple Aroclors may be quantitated and reported.

12.3. Second column confirmation of Aroclors will only be performed when requested by the client or regulatory program. The appearance of the multiple peaks in the sample usually serves as a confirmation of Aroclor presence. For Ohio VAP and DoD projects, both columns must meet criteria for positive detections.

12.4. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits, or if one is <10%.

Note: For Ohio VAP samples and DoD projects, all surrogates must meet acceptance limits, unless the surrogate is biased high and the sample is ND.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced in the main body of this SOP.

13.2. Method detection limits (MDL) are determined for all Aroclors.

14. POLLUTION PREVENTION

14.1. Refer to Section 14 of main body of this SOP.

15. WASTE MANAGEMENT

15.1. Refer to Section 15 of the main body of this SOP

16. REFERENCES

16.1. SW846, Update III, December 1996, Method 8082

16.2. SW846, Update IV, Revision 1, February 2007, Method 8082A

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Method 8082 and 8082A includes limited direction for congener specific quantitation. This is outside the scope of this SOP.

17.2. Tables

TABLE C1
Standard Analyte list and Reporting Limits for Methods 8082 and 8082A

	Reporting Limit, µg/L or µg/kg		
Compound	**Water**	**Soil**	**Waste**
Aroclor-1016	1.0	33	1000
Aroclor-1221	1.0	33	1000
Aroclor-1232	1.0	33	1000
Aroclor 1242	1.0	33	1000
Aroclor-1248	1.0	33	1000
Aroclor-1254	1.0	33	1000
Aroclor-1260	1.0	33	1000

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.
Ground water	1000 mL	5 mL
Low-level Soil	30g	10 mL
High-level soil / waste	1g	10 mL

TABLE C2
Instrumental Conditions for Methods 8082 and 8082A

Parameter	Recommended Conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	70°C for 0.5min, 30°C/min to 190°C, 2.5°C/min to 225, 18°C/min to 280°C, 3 min hold
Column 1	CLPesticide I, 30m, 0.53mm id, 0.5um
Column 2	CLPesticide II, 30m, 0.53 mm id, 0.5um
Injection	1µL
Carrier gas	Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

TABLE C3
Calibration Levels ug/mL for Methods 8082 and 8082A

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Aroclor 1016/1260	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1242	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1221 +1254	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1232	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1248	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1262	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1268	0.05	0.1	0.2	0.5	1.0	2.0
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	0.0025	0.005	0.01	0.025	0.05	0.1
Decachlorobiphenyl	0.0025	0.005	0.01	0.025	0.05	0.1

TABLE C4
Laboratory Control Sample (LCS) / Matrix Spike/Spike Duplicate (MS/MSD) and Surrogate Spike levels for Aroclor analysis µg/L or µg/kg for Methods 8082 and 8082A

	Aqueous	Soil	Waste
Aroclor 1016/1260	10	333	10,000
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

TABLE C5
Michigan Analyte List and Reporting Limits[1] for Methods 8082 and 8082A

	Reporting Limit		
Compound	**water (µg/L)**	**soil (µg/Kg)**	
Aroclor-1016	0.2	330	
Aroclor-1221	0.2	330	
Aroclor-1232	0.4	330	
Aroclor 1242	0.2	330	
Aroclor-1248	0.2	330	
Aroclor-1254	0.2	330	
Aroclor-1260	0.2	330	

[1] Reporting Limits are only for samples performed under the Michigan program.

TABLE C6
Suggested Analytical Sequence for Methods 8082 and 8082A

Initial Calibration

Injection #		
1	Solvent blank (optional)	
2	Aroclor 1016/1260	Level 1
3	Aroclor 1016/1260	Level 2
4	Aroclor 1016/1260	Level 3
5	Aroclor 1016/1260	Level 4
6	Aroclor 1016/1260	Level 5
7	Aroclor 1232	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
8	Aroclor 1242	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
9	Aroclor 1248	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
10	Aroclor 1221/1254	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
11	Aroclor 1268 or 1262	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
12	ICV	
13-32	Sample 1-20 (or as many samples as can be analyzed in 12 hours	
33	Aroclor 1016/1260	Level 3

Note: A solvent blank or primer may be analyzed at any time during the sequence when highly contaminated samples are expected. A solvent blank or primer may not be analyzed as routine immediately prior to standards.

12-hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Aroclor 1260 / 1016 mix. Mid-level standards of any other Aroclors expected to be present in the samples are also injected.

1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8151A is applied to the analysis of chlorinated phenoxy acid herbicides in extracts prepared by SOP NC-OP-031.

1.2. Table D1 lists compounds, which are routinely analyzed by this method and give the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

2. SUMMARY OF METHOD

2.1. This method presents conditions for the analysis of prepared extracts of phenoxy acid herbicides by gas chromatography. The herbicides, as their methyl esters, are injected onto the column, separated, and detected by electron capture detectors. Quantitation is by external standard methods.

3. DEFINITIONS

3.1. Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms and acronyms used in this document.

4. INTERFERENCES

4.1. Refer to the main body of this SOP for general information regarding chromatographic interferences.

4.2. Chlorinated acids and phenols cause the most direct interference with this method.

4.3. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples.

5. SAFETY

5.1. Refer to the main body of this SOP for general safety requirements.

5.2. All ^{63}Ni sources must be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.3. All ^{63}Ni sources must be inventoried every six months. If a detector is missing, the EH&S Director must be immediately notified and a letter sent to the NRC or local state agency.

6. EQUIPMENT AND SUPPLIES

6.1. A Ni_{63} electron capture detector is required.

6.2. Refer to Table D2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

6.4 Autosampler vials, inserts, and caps

7. REAGENTS AND STANDARDS

7.1. Refer to the main body of this SOP for general information on reagents and standards.

7.2. Refer to Table D4 for details of calibration standards. See the Standards Logbook for details on sample preparation.

7.3. Surrogate Standards

7.3.1. DCAA is the surrogate standard. Refer to Table D4 for details of surrogate standards.

8. SAMPLE PREPARATION, PRESERVATION, AND STORAGE

8.1. The holding time for herbicide extracts is 40 days from extraction to analysis. Samples must be refrigerated at $\leq 6^{\circ}C$.

9. QUALITY CONTROL

9.1. Refer to the main body of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes / spike duplicates (MS/MSD).

9.2. Refer to Table D3 for the components and levels of the Laboratory Control Sample (LCS) and matrix spikes / spike duplicates (MS/MSD) mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to the main body of this SOP for general calibration requirements.

10.2. Refer to Table D2 for recommended instrument operating conditions.

10.3. Calibration standards are prepared from purchased standards in the methyl ester form.

10.4. The low-level standard must be at or below the laboratory reporting limit.

10.5. The response for each analyte must be calculated by the procedures described in the main body of this SOP

10.6. Initial Calibration Verification (ICV)

10.6.1. An initial calibration verification (ICV) standard, from a second source, is analyzed immediately following the initial calibration. An acceptance criterion is ± 20%. If this is not met, a new initial calibration curve is analyzed.

10.7 Daily Continuing Calibration

10.7.1 A mid-level calibration mix is analyzed as the continuing calibration standard. At a minimum, this is analyzed after every 20 samples, including matrix spike / spike duplicate (ms/msd), Laboratory Control Sample (LCS), and method blanks. If 12 hours elapse, analyze the 12-hour standard sequence instead. If instrument drift is expected due to sample matrix or other factors, it may be advisable to analyze the continuing calibration standard more frequently.

11. PROCEDURE

11.1. Refer to the main body of this SOP for procedural requirements.

11.2. Extraction

11.2.1. The extraction procedure is described in SOP NC-OP-032.

11.3. Analytical Sequence

11.3.1. The analytical sequence starts with an initial calibration of at least five points, or a daily calibration that meets % difference criteria from an existing initial calibration.

11.3.2. The daily calibration must be analyzed at least once every 24 hours when samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence.

11.3.3. The daily calibration consists of mid level standards of all analytes of interest. Retention time windows must be updated with the daily calibration.

11.3.4. After every 12 hours a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest. Retention time windows are updated with continuing calibrations.

12. DATAANALYSISAND CALCULATIONS

12.1. Refer to the main body of this SOP for identification and quantitation of single component analytes.

12.2. The herbicides are analyzed as their methyl esters, but reported as the free acid. For this reason, it is necessary to correct the results for the molecular weight of the ester versus the free acid. This is achieved through the concentrations of the calibration standards. For example the 20µg/L calibration standard for 2,4-D contains 21.3 µg/L of the methyl ester. No further correction is necessary.

12.3. A routine 10X dilution occurs on final extracts for all samples. Due to a LIMS limitation, the dilution factor field in LIMS cannot be used when a dilution is routine, because the dilution factor is automatically applied to all reference values creating reporting problems. For the herbicide analysis, the extract volume will be 10mL and an aliquot at 10X dilution will be analyzed. The final extract volume recorded on the laboratory bench sheet will be recorded as 100mL to avoid using the dilution factor field in LIMS.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced in the main body of this SOP.

14. POLLUTION PREVENTION

14.1. Refer to Section 14 of the main body of this SOP.

15. WASTE MANAGEMENT

15.1. Refer to Section 15 of the main body of this SOP.

16. REFERENCES

16.1. Method 8151A, SW-846, Update III, December 1996

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. None

17.2. TABLES

TABLE D1
Standard Analyte List for Method 8151A

Compound	CAS Number	Reporting Limit, µg/L or µg/kg		
		Aqueous	Soil	Waste
2,4-D	94-75-7	4	80	4000
2,4-DB	94-82-6	4	80	4000
2,4,5-TP (Silvex)	93-72-1	1	20	1000
2,4,5-T	93-76-5	1	20	1000
Dalapon	75-99-0	2	40	2000
Dicamba	1918-00-9	2	40	2000
Dichloroprop	120-36-5	4	80	4000
Dinoseb	88-85-7	0.6	12	600
MCPA	94-74-6	400	8000	400,000
MCPP	93-65-2	400	8000	400,000
Pentachlorophenol				

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.	Dilution Factor
Ground water	1000 mL	10 mL	10
Low-level Soil without GPC	30g	10 mL	10
High-level soil / waste	1g	10 mL	10

Specific reporting limits are highly matrix dependent. The reporting limits listed above are provided for guidance only and may not always be achievable. For special projects, the extracts may be analyzed without any dilution, resulting in reporting limits 20 times lower than those in Table D1.

TABLE D2
Recommended Instrumental Conditions for Method 8151A

PARAMETER	Recommended conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	80,2/30/170,0/1/180,1
Column 1	CLPesticideI, 30m, 0.53 mm id, 0.5 um
Column 2	CLPesticide II, 30m, 0.53 mm id, 0.5um
Injection	1-2µL
Carrier gas	Hydrogen
Make up gas	Nitrogen

Recommended conditions must result in resolution of all analytes listed in Table D1.

The reporting limits listed in Table D1 will be achieved with these calibration levels and a 20-fold dilution of the sample extract. Lower reporting limits can be achieved with lesser dilutions of the sample extract.

TABLE D3
Laboratory Control Sample (LCS)/Matrix Spike/Spike Duplicate (MS/MSD) and Surrogate Spike levels µg/L or µg/kg[1] for Method 8151A

	Aqueous	Soil	Waste
2,4-D	40	400	20000
Silvex	10	100	5000
2,4,5-T	10	100	5000
2,4-DB	40	400	20000
Dalapon	20	200	10000
DCAA (surrogate)	40	400	20000
Dicamba	20	200	10000
MCPP	4000	40000	200000
MCPA	4000	40000	200000
Dichloroprop	40	400	2000
Pentachlorophenol	5	50	2500
Dinoseb	6	60	300

[1] Laboratory Control Sample (LCS), MS and SS spikes are as the free acid.
Note: Dinoseb is a poor performing analyte. No corrective action will be taken if recovery is outside acceptance limits.

TABLE D4
Calibration Levels for Methods 8151A (ng amount)

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
2,4 D	0.02	0.04	**0.08**	0.16	0.32	0.64
2,4 DB	0.02	0.04	**0.08**	0.16	0.32	0.64
2,4,5 T	0.005	0.01	**0.02**	0.04	0.08	0.16
2,4,5 TP (Silvex)	0.005	0.01	**0.02**	0.04	0.08	0.16
Dalapon	0.01	0.02	**0.04**	0.08	0.16	0.32
DCAA (surr)	0.02	0.04	**0.08**	0.16	0.32	0.64
Dicamba	0.01	0.02	**0.04**	0.08	0.16	0.32
Dichloroprop	0.02	0.04	**0.08**	0.16	0.32	0.64
Dinoseb	0.003	0.006	**0.012**	0.024	0.048	0.096
MCPA	2	4	**8**	16	32	64
MCPP	2	4	**8**	16	32	64
Pentachlorophenol	0.0005	0.005	**0.01**	0.02	0.04	0.08

Bold levels indicate CCV

1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8015B or 8015C is applied to the determination of the concentration and **tentative** identification of extractable petroleum (diesel range) hydrocarbon mixes in waters, wastewaters, soils, and sludges. This Appendix is applicable to extracts derived from any matrix which are prepared according to the appropriate sample extraction SOPs.

1.2. Table E2 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed. Other analytes may be analyzed by this method if the quality control criteria in Section 9 and the initial demonstration of method performance in Section 13 are met. Reporting limits are also listed in Table E2. The laboratory carbon range for Ohio VAP and BUSTR projects is Middle Distillates (C10-C20) and Heavy Distillates (C20-C34).

2. SUMMARY OF METHOD

2.1 This method presents conditions for the analysis of total petroleum hydrocarbons by gas chromatography. The total petroleum hydrocarbon samples are injected into the column, separated, and detected by flame ionization detectors (FID). Quantitation is by external standard methods.

3. DEFINITIONS

3.1. Refer to the TestAmerica North Canton Quality Assurance Manual (QAM), current version, for definitions of terms and acronyms used in this document.

4. INTERFERENCES

4.1. Refer to the main body of this SOP for general information regarding chromatographic interferences.

4.2. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples.

5. SAFETY

5.1. Refer to the main body of this SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

6.1. A flame ionization detector (FID) is required.

6.2. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

6.3. Autosampler vials, inserts, and caps

7. REAGENTS AND STANDARDS

7.1. Refer to the main body of this SOP.

7.2. The petroleum hydrocarbons (Diesel Fuel) are purchased from a chemical supplier when available. When no chemical supplier is available, the fuels are purchased from public sources. See the Standards Logbook for details on sample preparation.

7.3. Refer to Table E3 for details of calibration standards.

7.4. Surrogate Standards

7.4.1. Nonane (C9) is the surrogate standard.

8. SAMPLE PREPARATION, PRESERVATION, AND STORAGE

8.1. The holding time for semivolatile extracts is 40 days from extraction to analysis. Samples must be refrigerated at <6°C.

9. QUALITY CONTROL

9.1. Refer to the main body of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes / spike duplicates (MS/MSD).

9.2. Matrix spikes / spike duplicates (MS/MSD) recoveries are calculated from a diesel calibration.

9.3. Surrogates

9.3.1. Because of the nature of the TPH analysis, - certain petroleum mixtures can override the C9 (Nonane) surrogate.

Note: Ohio VAP rules require reanalysis when surrogate recoveries are outside of control limits. Re-extraction is required if surrogates are outside of control limits.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to the main body of this SOP for general calibration requirements. The low-level calibration standard must be at, or below, the reporting limit.

10.2. Refer to Table E1 for recommended instrument conditions.

10.3. Initial Calibration

10.3.1. Prior to the initial calibration, a marker solution consisting of alkanes from C10-C44 is analyzed, If additional carbon ranges are needed, a separate solution with alkanes from C10-C50 can be analyzed with a modified instrument program. The marker solution establishes the retention time window.

10.3.2. Analyze a diesel calibration calibration, using a minimum of five points, referring to the recommended instrument conditions. The calibration concentrations are 100, 200, 500, 1000, and 2000 ng/uL. A 5000ng/uL standard may be analyzed if needed. The retention time window of C10-C32 must be used for the Diesel calibration. The low-level standard must be at or below the reporting limit. The other standards define the working range of the detector.

Note: For special projects, retention time windows can be customized to reflect additional carbon ranges. The additional carbon ranges are quantitated against the diesel calibration.

10.4. Initial Calibration Verification (ICV)

10.4.1. An initial calibration verification (ICV) standard, from a second source, is analyzed immediately following the initial calibration. An acceptance criterion is ± 20%. If this is not met, a new initial calibration curve is analyzed.

10.5. Daily Continuing Calibration

10.5.1. Refer to Section 10 of the Method 8000B section of this SOP for general calibration requirements.

10.5.2. A mid-range standard of diesel, C10-20, and C20-34 is used, as appropriate, for the CCV. The acceptance criterion is ±15%. This marker solution must be analyzed at the beginning of each sequence. For Method 8015C, the CCV acceptance criteria is ± 20%

11. PROCEDURE

11.1. Refer to the main body of this SOP for procedural requirements.

11.2. A suggested analytical sequence is given in Table E4.

11.3. Petroleum Hydrocarbon Identification and/or Fingerprinting

11.3.1. To identify the type of petroleum hydrocarbon, compare the chromatographic peak pattern to the patterns of known petroleum hydrocarbons analyzed under identical chromatographic conditions. Samples are quantified against diesel, but fingerprinting may be done when client requested.

11.3.2. Positive matching may not be possible, even using site-specific hydrocarbons. Degradation of the pattern can occur during environmental exposure of the fuel. See Table E2 for possible fingerprints.

11.3.3. Samples are quantified against the initial calibration of diesel or DRO on a single column.

11.3.4. The total height or area of the hydrocarbon is determined in the same manner used for the hydrocarbon standard.

11.3.5. If the amount of sample injected into the GC exceeds the working range of the calibration curve, an appropriate dilution is performed before reanalysis.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the main body of this SOP for identification and quantitation of single component analytes.

12.2. Surrogate recovery results are calculated and reported for Nonane (C-9). The surrogate must be within QC criteria. Corrective action is only necessary if Nonane (C-9) is outside of acceptance limits, unless the surrogate is high and the sample is ND.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced in the main body of this SOP.

14. POLLUTION PREVENTION

14.1. Refer to Section 14 of the main body of this SOP.

15. WASTE MANAGEMENT

15.1. Refer to Section 15 of the main body of this SOP.

16. REFERENCES

16.1. SW846, Method 8015B, Nonhalogenated Organics Using GC/FID, Test Methods for Evaluating Solid Waste, Third Edition, USEPA

16.2. SW846, Method 8015C, Nonhalogenated Organics by Gas Chromatography, Test Methods for Evaluating Solid Waste, Revision 3, February 2007.

TABLE E1
Recommended Instrument Conditions for Methods 8015B and 8015C

Parameter	Recommended Conditions
Column	RTX-5
Initial Temperature	40°C
Initial Hold Time	4 minutes
Temperature Program	10°C/minute
Final Temperature	280°C
Final Hold Time	10 minutes
Injection	1µL
Carrier Gas	Hydrogen carrier gas - flow rate 5 - 6 mL/min
Detector Gas Mixture	Air hydrogen mixture in a 10:1 ratio, air 80 - 120 mL/min, hydrogen 8 -12 mL/min

TABLE E2
Reporting Limits for TPH Analysis

Analyte	Reporting Limits		
	Water (µg/L)	Solids (mg/kg)	Waste Dilution (mg/kg)
TPH (as Diesel) or DRO	500	16.7	200
C10-C20 (OVAP & BUSTR - Middle Distillates)	500	16.7	
C20-C34 (OVAP & BUSTR – Heavy Distillates)	500	16.7	
Fingerprint Compounds[1]			
Mineral Spirits	Kerosene	Motor Oil	
Hydraulic Oil	Jet Fuel	Stoddard Solvent	

[1] This list represents most of the common petroleum hydrocarbons. The list may be expanded to include other petroleum hydrocarbons.

TABLE E3
Calibration Levels for Methods 8015B and 8015C (ng/L)

	Level 1	Level 2	Level 3	Level 4	Level 5
TPH (as Diesel)	100	200	500	1000	2000
TPH (as Diesel) LVI [1]	20	40	100	200	400

[1] For LVI analysis, a 5 ul injection will be used.

TABLE E4
Suggested Analytical Sequence for Method 8015B and 8015C

Initial Calibration

Solvent blank (optional)
Primer if needed
Marker Solution
Diesel Standard All levels
ICV
Up to 20 samples unless 12 hours comes first)
Mid level Diesel Standard (Continuing calibration)

Note: A solvent blank or primer may be analyzed at any time during the sequence when highly contaminated samples are expected. A solvent blank or primer may not be analyzed as routine immediately prior to standards.

Note: The initial primer is used if the instrument has been idle for a period of time.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Canton

SOP No. NC-GC-032, Rev. 5
Effective Date: 04/29/13

Title: ANALYSIS OF DISSOLVED GASES IN GROUNDWATER

[Method: Method RSK-175]

Approvals (Signature/Date):

[redacted] 04/29/13 Technology Specialist Date	04/29/13 Health & Safety Coordinator Date
04/29/13 Quality Assurance Manager Date	[redacted] 04/29/13 Laboratory Director Date

This SOP was previously identified as SOP NC-GC-032, Rev 4, dated 05/06/10

Facility Distribution No. _____ **Distributed To:_____**

TABLE OF CONTENTS

1. SCOPE AND APPLICATION

1.1 This document describes a procedure for the determination of dissolved gases in groundwater. The method is applicable to the preparation of water samples for the analysis of the headspace to quantify part-per-billion levels of dissolved gases in water samples.

1.2 This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. A water sample is collected in the field in a 43-mL VOA vial with no headspace. Prior to analysis, the sample is transferred into a 21-mL serum vial with a crimp cap. Headspace is generated using UHP helium fortified with surrogate compounds. The sample is loaded onto the headspace autosampler and analyzed by a Gas Chromatograph (GC) equipped wth a Flame Ionization Detector (FID)

3. DEFINITIONS

3.1 Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current version. See Table 3 at the end of this document for a glossary of acronyms.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review

the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4. It is recommended that analysts break up work tasks to avoid repetitive motion tasks, such as opening a large number of vials or containers in one time period.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with a sticker that reads "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and to a Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1 Sample Containers:

6.1.1 Unpreserved 43mL VOA vials

6.1.2 Pre-preserved 43 mL VOA vials containing reagent grade or equivalent HCl

6.1.3 21 mL crimp cap vials

6.2 Instrumentation

6.2.1 Column (FID - Agilent PLOT-Q; 30m, 0.53mm ID

6.2.2 Autosampler - Agilent 7694 Headspace autosampler

6.3 Syringes - 10μL - 5.0-mL gas tight syringes with 22 gauge side port needles.

6.4 Relief needles: 18 gauge with side port for use in relieving pressure created during headspace formation.

6.5 Data System

7. REAGENTS AND STANDARDS

7.1. Ultrahigh purity helium is used to create headspace in initial calibration vials.

7.2. Calibration Standards: The primary standard is purchased through Matheson Tri Gas. The calibration standard is comprised of two cylinders--one composed of nominally 0.1% (mole basis) methane, ethane, ethylene, acetylene, propane, propene, and 2-Methylpropane; and the second composed of nominally 1% methane, ethane, ethylene, acetylene, propane, propene, and 2-Methylpropane. The second source standard is 1% mix provided by Scott Specialty Gases.

7.3. Surrogate Calibration Standard: The surrogate standard is purchased though Matheson Tri Gas. The standard contains 5% 1,1,1-trifluoroethane in UHP helium.

7.4. Surrogate fortified helium: The surrogate mixture is purchased through Matheson Tri Gas. The standard contains UHP helium fortified with 0.325% 1,1,1-trifluoroethane. The surrogate mixture is used to create headspace in all samples other than the initial calibration.

7.5. Reagent Water. High purity water that meets the requirements for a method blank when analyzed. Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight.

7.6. The calibration levels are achieved by using a syringe to inject different amounts of the standard into a 21 mL vial of reagent water. A final volume of 4 mL headspace is created in each vial (see Table 10.1).

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Samples are collected in the field in a 43-mL screw cap VOA vial. The samples are preserved with 1:1 HCl to a pH of less than 2. Care should be taken that no headspace is present when capping the vials. A minimum of three vials per analysis is required. Double the volume for a sample with an MS/MSD.

8.2 Samples are maintained at a temperature of 4°C ± 2°C and should be analyzed within 14 days of collection.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, and MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents, the same processes, and the same personnel.

9.2. Method Blank (MB)

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water, and all other reagents specific to the method. The method blank is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit, with the exception of common laboratory contaminants.

9.2.2. Corrective Action for Blanks

9.2.2.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are re-prepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.2.2. If there is no analyte greater than the RL in the samples associated with an impacted method blank, or the impacted analyte(s) in the sample are 10X the concentration found in the blank, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One LCS from an independent source must be processed with each preparation batch. The LCS is fortified to the concentration of a standard near the midpoint of the calibration curve. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence

that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. Corrective Action for LCS

9.3.2.1. If any analyte is outside established control limits, the system is out of control and corrective action must occur.

9.3.2.2. The only exception is if the LCS recoveries are biased high and the associated sample is ND for the analyte(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

9.3.2.3. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair is processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. Corrective action for MS/MSDs

9.4.2.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.4.2.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported and flagged with a "4" in LIMS.

9.4.2.3. If client program requirements specify to confirm matrix interference's, repreparation and reanalysis of the MS/MSD may be necessary.

9.5. Surrogate

9.5.1. 1,1,1-Trifluoroethaneis the surrogate for RSK analytes.

9.6. Control Limits

9.6.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.6.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are accessible via LIMs

9.7. Method Detection Limits (MDLs) and MDL Checks

9.7.1. MDLs and MDL Checks are established by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.7.2. MDLs are accessible via LIMs

9.8. Nonconformance and Corrective Action

9.8.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action.

10. CALIBRATION AND STANDARDIZATION

10.1 Initial Calibration – Establish an initial calibration curve using the concentrations noted in the table below.

Mix 1 = Low-level calibration standard

Mix 2 = High-level calibration standard

Concentrations are in µg/L unless noted otherwise.

Compound	Level 1 Mix 1 4 mL Helium	Level 2 Mix 1 4 mL Helium	Level 3 Mix 1 3.8 mL Helium	Level 4 Mix 1 3.4 mL Helium	Level 5 Mix 2 3.3 mL Helium	Level 6 * Mix 2 2.8 mL Helium	Level 7 Mix 2 1.9 mL Helium	Level 8 Mix 2 2 mL Helium	Level 9 Mix 2 No Helium
Volume Injected (uL)	6.5	25	100	300	100	300	900	2000	4000
Methane	0.25	.96	3.86	11.58	38.58	115.73	347.19	771.57	1543.06
Ethane	0.47	1.81	7.26	21.78	72.58	217.73	653.19	1451.53	2903.06
Ethene	0.44	1.69	6.78	20.33	67.75	203.26	609.78	1355.06	2710.12
Acetylene	0.41	1.57	6.27	18.81	62.69	188.06	564.19	1253.76	2507.53
Propane	0.69	2.66	10.64	31.91	106.34	319.01	957.02	2126.71	4253.41
2-Methylpropane	0.91	3.51	14.01	42.04	140.09	420.26	1260.79	2801.76	5603.53
Propene	0.66	2.54	10.15	30.46	101.51	304.54	913.61	2030.24	4060.47
Surrogate Calibration Standard (5% Trifluorethane)									
Volume Injected (uL)	6.5	25	100	300	600	900	1200	–	–
1,1,1-Trifluoroethane surrogate	65.83	253.17	1012.69	3038.08	6076.16	9114.25	12152.33	–	–

* This level is used for CCVs also.

10.1.1. Preparation of Initial Calibration Standards

10.1.1.1. Fill a vial with purged reagent water and cap, leaving no headspace in the vial.

10.1.1.2. Using the calibration volumes from the table above, withdraw UHP helium from a cylinder using a gastight syringe. Place a relief needle into the headspace vial and inject the helium while keeping the vial inverted. The helium will displace water through the relief needle. Calibration gas can now be added to the still-inverted headspace vial.

10.1.1.3. Once pressure equilibrium has been reached, the relief needle is removed. The vial can be turned upright and left to equilibrate for two hours. After two hours, the vials are placed in the auto sampler rack for analysis.

10.1.2. For each analyte, calculate the mean calibration factor (CF) from analyses of the calibration solutions.

10.1.3. Calculate the standard deviation (SD) and relative standard deviation (RSD) from each mean.

10.1.4. The percent RSD average of all analytes must be $\leq$ 30%.

10.1.5. Removal or replacement of levels from the middle of a calibration (i.e., levels other than the highest or lowest) is not permitted unless an injection or instrument problem confined to that point can be clearly documented as described below.

10.1.6. If the analyst can document that a level is not valid because of an injection or instrument problem confined to that run, the level may be excluded if the curve still has sufficient levels, or the run may be repeated once only. The whole level (all compounds) must be removed or replaced. The curve is evaluated with the level removed or replaced. If the curve still fails to meet criteria, then corrective action must be taken and the whole curve re-analyzed. Corrective action may include, but is not limited to, instrument maintenance and/or re-preparation of standards.

10.1.7. One of the following conditions must be satisfied to allow removal or replacement of a level.

- The data file is corrupted and unusable or the run is interrupted before completion.

- The analyst observes and documents a problem such as leaking of a purge vessel.

- For external standard methods, the average amount of analyte recovered is less than 70% or greater than 130% of the expected value.

10.1.8. The reason for replacing the level **must** be documented in the run log. The fact that the curve passes criteria with the level removed is **not** alone sufficient evidence to document an injection or instrument problem confined to the level.

10.1.9. Removal of the highest or lowest levels is permitted, but the calibration range must be adjusted accordingly. If the lowest level is removed, then the reporting limit is raised to be equivalent to the lowest level used in the calibration curve. In any event, the number of levels remaining in the calibration must be at least that required by the method.

10.1.10. Removal of the highest or lowest point is permitted on a compound specific basis. This may be necessary when strongly responding and poorly responding analytes are included in the same standard mix at the same level. Each compound must have at least the minimum number of calibration levels required by the method.

10.2. Continuing Calibration Verification (CCV) – Analyze a CCV at the beginning of each 24-hr analytical window. The %D between the CCV CF and the calibration average CF for each analyte must be less than or equal to 30%.

10.2.1. Withdraw 3.7 mL surrogate fortified helium from a cylinder using a gastight syringe. Place a relief needle into the headspace vial and inject the helium while keeping the vial inverted. The helium will displace water through the relief needle. Calibration gas can now be added to the still-inverted headspace vial.

10.2.2. Add 300µL of standard mix 2 to the inverted vial. Once pressure equilibrium has been reached, the relief needle is removed. The vial can be turned upright and left to equilibrate for two hours.

10.3. Initial Calibration Verification - An ICV must be analyzed run following the acquisition of the initial calibration. The calibration factor (CF) for the ICV must be within 30%D of that from the initial calibration. The ICV is prepared in the same manner as the CCV, using 300 µL of the second source standard.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.3. Sample Analysis

11.3.1. Sample Analysis (GC-FID) - Remove the samples from the refrigerator. Immediately pour the sample into a 21 mL vial and seal with a crimp cap, taking care to avoid headspace. Allow the vial to come to room temperature. This will take at least one hour. Insert an 18 gauge relief needle into the septum. Using a 5 mL gastight syringe, inject 4 mL of surrogate fortified helium into the sample. The helium forces out an equal amount of sample through the relief needle to create a headspace volume of 4 mL. Withdraw the needle and syringe from the vial, then set vials upright and allow samples to equilibrate for two hours. Load the sample onto the headspace autosampler. 1mL of the sample headspace is injected directly onto the GC column where the target compounds, if present, are detected by FID. Acquire the data and process on Chrom. The recommended instrument operating conditions are outlined below.

Recommended GC Conditions

12.0 constant pressure
Oven Program: 50 for 1.5 minutes
25°C/min to 200°C hold 0.5 min

Recommended FID Conditions

FID Temp:	250°C	Hydrogen Flow:	40 mL/min
Air Flow:	400 mL/min		
Helium Makeup:	45 mL/min		

Recommended Headspace Auto sampler Conditions

Zone Temp	50°C	Vial Pressurization time	0.1 min
Sample Loop Temp	100°C	Sample Loop fill time	0.05 min
Transfer Line Temp	110°C	Sample Loop Equil. time	0.01 min
GC Cycle time	12 min	Sample Injection time	0.30 min
Vial equilibration time	1.3 min		

11.3.2. QC sample prep.

11.3.2.1. The method blank is prepared as a sample.

11.3.2.2. An LCS is prepared like an ICV, using 300 µL second source standard after 3.7 mL of headspace has been created..

11.3.2.3. An MS/MSD pair is prepared as a regular sample, but 300 µL of second source standard is injected after 3.7 mL of headspace has been created.

11.4. Analytical Documentation

11.4.1. Record all analytical information in LIMS, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.4.2. All standards are logged into the LIMS standards and reagents module. All standards are assigned a unique number for identification.

11.4.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.4.4. Sample results and associated QC are uploaded directly into LIMS Level I and Level II reviews are is done in LIMS

12. DATA ANALYSIS AND CALCULATIONS

12.1 Calibration Factor for GC-FID

$$CF = A/C$$

Where:

CF	=	Calibration factor of an analyte
A	=	Instrument response (peak area or height)
C	=	Concentration of an analyte in sample

12.2 Percent Difference for Calibration Factors

$$\%\ D = (Avg_{CF} - CF / Avg_{CF}) \times 100$$

Where:

Avg_{CF}	=	Average CF for an analyte from the initial calibration
CF	=	CF for an analyte from standard

12.3 Relative Standard Deviation

$$\%RSD = (SD / Avg_{CF}) \times 100$$

Where:

Avg_{cf}	=	Average CF for an analyte from the initial calibration
SD	=	Standard Deviation of CFs for a compound

12.4 Sample Concentration in water

$$C = (A/Avg_{CF}) \times DF$$

Where:

C = Concentration of target analyte in sample
A = Instrument response (peak area or height) Avg_{CF} = Average CF for an analyte from the calibration
DF = Dilution factor

12.5 Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generate (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method

15.2.1. All sample vials are collected in boxes and removed from the lab to storage. The Waste Coordinator handles crushing the vials and proper disposal

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks followed by annual refresher training.

16. REFERENCES

16.1. References

16.1.1. RSK SOP-175, Revision 0, August 11, 1994

16.1.2. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.3. Corporate Quality Management Plan (CQMP), current version

16.1.4. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.5. Revision History

Historical File:		Revision 0: 05/13/02		
		Revision 1: 11/16/04		
		Revision 2: 02/01/07		
		Revision 3: 03/21/08		
		Revision 4: 05/06/10		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-016

16.2.6. Standards and Reagents,NC-QA-017

16.2.7. Acceptable Manual Integration Practices, CA-Q-S-002

16.2.8. Calibration Curves (General), CA-Q-S-005

16.2.9. Selection of Calibration Points, CA-T-P-002

17. MISCELLANEOUS(TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. The reporting limit for all analytes is 1 ug/L. Check reporting limits in TALS

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.1.3. Compound Constants

Table 2
COMPOUND CONSTANTS

Compound	Molecular Weight (g)
Methane	16
Ethane	30
Ethene	28
Acetylene	26
Propane	44
2-Methylpropane	58
Propene	42
1,1,1-Trifluoroethane (Surrogate)	84

Table 3

EXAMPLE CALCULATION

(Calculations Based on 22°C at 754 mmHg – Molar Equivalent .04099)

(Level 8) 2,000 ul of 10,000ppmv – 17 mL H_20

$$\frac{10{,}000\ ul\ CH_4}{L\ He} \times \frac{1\ L\ CH_4}{1{,}000{,}000\ ul\ CH_4} \times \frac{.04099\ moles\ CH_4}{1\ LCH_4} \times \frac{16\ g\ CH_4}{1\ mole\ CH_4} \times \frac{.002\ L\ He}{.017\ LH_2 0} \times$$

$$\frac{1{,}000{,}000\ ug\ CH_4}{1\ g\ CH_4} = \frac{13.1168}{.017} = 771.57 \frac{ug\ CH_4}{LH_2 0}$$

EXPLANATION OF TERMS:

$\frac{10{,}000\ ul\ Methane}{L\ Helium}$ = Puts PPMV methane into volumetric ratio per liter helium. (Helium is the gas used to generate head space in the sample vial.)

$\frac{1\ L\ Methane}{1{,}000{,}000\ ul\ Methane}$ = Simple units conversion.

$\frac{.04099\ moles\ Methane}{L\ Methane}$ = Molar equipment of methane at standard temperature and pressure per ideal gas law.

$\frac{16\ g\ Methane}{1\ mole\ Methane}$ = Molar weight of methane.

$\frac{.002\ L\ Helium}{.017\ LH_2 0}$ = Amount of helium in calibration level #8 standard distributed over 17 ml of reagent water in sample vial.

$\frac{1{,}000{,}000\ ug\ Methane}{1\ g\ Methane}$ = Units conversion from grams to µg of methane to arrive at µg/L concentration of methane.

$\frac{13.1168}{.017}$ = Collection of terms.

$771.57 \mu g\ L^{-1}$ Methane = Final concentration of methane in calibration level #8.

Table 4
GLOSSARY OF ACRONYMS

CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CF	Correction Factor
COC	Chain of Custody
CQMP	Corporate Quality Management Plan
DOC	Demonstration of Capability
DOD	Department Of Defense
EH&S	Environmental Health and Safety
FID	Flame Ionization Detector
GC	Gas Chromatography
ICB	Initial Calibration Blank
ICV	Initial Calibration Verification
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
MB	Method Blank
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheet
NCM	Non Conformance Memo
OSHA	Occupational Safety and Health Administration

Table 4
GLOSSARY OF ACRONYMS

PEL	Permissible Exposure Limit
PTFE	Polytetrafluorothylene
QAM	Quality Assurance Manual
QA/QC	Quality Assurance/Quality Control
RF	Response Factor
RPD	Relative Percent Difference
SOP	Standard Operational Procedure
STEL	Short Term Exposure Limit
TWA	Time Weighted Average
UHP	Ultra High Purity
VOA	Volatiles

TestAmerica Canton

SOP Amendment Form

SOP NUMBER: NC-MT-012 Rev. 4

SOP TITLE: Inductively Coupled Plasma – Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis

REASON FOR ADDITION OR CHANGE: Updating IS limits

CHANGE EFFECTIVE FROM: (DATE): Latest signature date below

Change(s) Made: Sections 11.5.5.1 and 11.5.5.2 have been changed to read:

If the internal standard counts fall within ±~~30%~~ 50% of the counts observed in the ICB or calibration blank then the data is acceptable.

If the internal standard counts in the field samples are more than ± ~~30%~~50% higher than the expected level, a dilution is needed due to matrix interference.

EDITED BY/DATE: MFG 12/12/13

*APPROVED BY:

Technical Reviewer Signature Date: 12/12/13

QA Manager Signature Date: 12/12/13

Technical Director Signature Date: 12/13/13

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Canton

SOP No. NC-MT-012, Rev. 4
Effective Date: 09/13/13

Title: INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSES

[Methods: SW846 Methods 6010B, 6010C, and EPA Method 200.7]

Approvals (Signature/Date):

Technology Specialist	09/10/13 Date	Health & Safety Coordinator	09/06/13 Date
Quality Assurance Manager	09/13/13 Date	Laboratory Director	09/05/13 Date

This SOP was previously identified as SOP NC-MT-012, Rev 3-A, dated 04/17/12

Facility Distribution No. _____ **Distributed To: _____**

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LIST OF APPENDICES:

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma -Atomic Emission Spectroscopy (ICP-AES) using SW-846 Methods 6010B, 6010C, and EPA Method 200.7. Table I of Appendix A lists the elements appropriate for analysis by Methods, 6010B, 6010C, and 200.7. Additional elements may be analyzed under Methods, 6010B, 6010C, and 200.7 provided that the method performance criteria presented in Section 13.0 are met.

1.2. ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity, and optimum concentration ranges of the metals will vary with the matrices and instrumentation used.

1.3. Methods 6010B and 6010C are applicable to the determination of dissolved, suspended, total recoverable, and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, biological, and TCLP, EP, and other leachates/extracts. All matrices require digestion prior to analysis. Silver concentrations must be below 2.0 mg/L in aqueous samples and 100 mg/kg in solid matrix samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data.

1.4. Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable, and total elements in water, waste water, and solid wastes. All matrices require digestion prior to analysis. Silver concentrations must be below 0.1 mg/L in aqueous samples.

2. SUMMARY OF METHOD

2.1. This method describes a technique for the determination of multi elements in solution using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by radio frequency inductively-coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences must also be recognized and appropriate actions taken.

Alternatively, multivariate calibration methods may be chosen for which point selection for background correction is superfluous since whole spectral regions are processed.

2.2. Refer to NC-IP-010, Acid Digestion of Soils by SW846 Method 3050B, and NC-IP-011, Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods, for details on sample preparation methods.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current version for additional definitions. Refer to Appendix B for a cross reference of method definitions.

4. INTERFERENCES

4.1. Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.
- Stray light from the line emission of high concentration elements.

4.1.1. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.1.2. Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections IECs must be applied to the analyte to remove the effects of these unwanted emissions.

4.1.3. Physical interferences are generally considered to be effects associated with sample transport, nebulization, and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects) at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in

viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump, mass flow controller, use of an internal standard, and/or use of a high solids nebulizer can reduce the effect. Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4-ppm STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent

			eye damage.
1 – Always add acid to water to prevent violent reactions			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3.1. The plasma emits strong UV light and is harmful to vision. **NOTE**: **AVOID looking directly at the plasma**

5.3.2. The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers must not go near the instrument while in operation.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Metals digestates can be processed outside of a fume hood. Solvent and waste containers must be kept closed unless transfers are being made.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler and background correction.

6.2. Radio Frequency Generator

6.3. Argon gas supply, welding grade or equivalent

6.4. Coolflow or appropriate water cooling device

6.5. Peristaltic Pump

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes – ranging from 5 µL → 10 ml

6.7. Class A volumetric flasks – range from 50 ml → 2000 ml

6.8. Autosampler tubes

7. REAGENTS AND STANDARDS

7.1 Intermediate standards are purchased as custom multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles. Intermediate standard solutions must be

replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Expiration dates can be extended provided that the acceptance criteria described in laboratory-specific SOPs are met. Additional information can be found in SOP NC-QA-017. Standard or spiking concentrations, as well as vendors, are subject to change.

7.2 Working calibration, calibration verification solutions, and internal standard solutions must be prepared in a matrix of 5% hydrochloric and 5% nitric acids. Refer to Tables II, III, and IV (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification, interference correction, and spiking solutions. Refer to the laboratory Standard Logbook or Reagent Logbook for details on standard or reagent preparation.

7.3 Concentrated nitric acid (HN_{O3}), trace metal grade or better.

7.4 Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.5 Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron is to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. Soil samples do not require preservation, but must be stored at 4°C ± 2° until the time of preparation.

8.4. Metals samples that are preserved at the laboratory must be held for 24 hours before digestion.

Note: If the samples are preserved the same day of collection, the 24-hour waiting period is not required

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. Prior to analysis of any analyte using Methods 200.7, 6010B, or 6010C, the following requirements must be met.

9.1.2. Instrument Detection Limit (IDL): The IDL for each analyte must be determined for each analyte wavelength used for each instrument. The IDL

must be determined annually for client-specific projects. For DoD work, refer to SOP NC-QA-016. If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be re-determined. The IDL will be determined by multiplying by 3, the standard deviation obtained from the analysis of a blank solution, with seven consecutive measurements. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples).

9.1.3. Instrument Detection Limits (IDLs), Method 6010C: IDLs are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the lower limit of quantitation, nor should they be used in establishing this limit. It may be helpful to compare the calculated IDLs to the established lower limit of quantitation; however, it should be understood that the lower limit of quantitation needs to be verified using the criteria in Section 10.7.

9.1.4. Method Detection Limit (MDL): An MDL must be determined for each analyte prior to the analysis of any client samples. Refer to TestAmerica Canton SOP NC-QA-021 and CA-Q-S-006 for details on MDL analysis and criteria.

9.1.5. Linear Range Verification (LR): The linear range must be verified every six months for each analyte wavelength used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample. The standards used to verify the linear range limit must be analyzed during a routine analytical run, and must read within 10% of the expected value.

9.1.5.1. For the **initial** determination of the upper limit of the linear dynamic range (LDR) for each wavelength, determine the signal responses from a minimum of three to five different concentration standards across the estimated range. One standard must be near the upper limit of the estimated range. The concentration measured at the LDR must be no more than 10% less than the expected level extrapolated from lower standards. If the instrument is adjusted in any way that may affect the LRs, new dynamic ranges must be determined. The LR data must be documented and kept on file.

9.1.6. Background Correction Points: To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line inter-element spectral interference, or a computer routine must be used for automatic correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the ICP instrument manual for specific procedures to be used in setting

background correction points.

9.1.7. Inter-element Corrections (IECs): ICP inter-element correction factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be re-determined. When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC, then the possibility of contamination must be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., ICP-MS). Published wavelength tables (e.g., MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs. Refer to the instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any inter-element interference which results in a false analyte signal greater than ± the RL. For elements with a reporting limit of 10 µg/L or less, the signal must be ± two times the RL. To determine IECs, run a single element standard at the established linear range. To calculate an IEC, divide the observed concentration of the analyte by the actual concentration of the "interfering element."

Note: Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the Trace as reflected by the ICSA response. Additional spectral interference is present from easily ionizable elements such as potassium and sodium in axial viewing instruments.

9.1.8. Rinse Time Determination: Rinse times must be determined upon initial set-up of an ICP instrument. To determine the appropriate rinse time for a particular ICP system, the linear range verification standard (see Section 9.1.4) must be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system. For some analytes, it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). Until the required rinse time is established, the method recommends a rinse period of at least 60 seconds between samples and standards. If a memory effect is suspected, the sample must be re-analyzed after a rinse period of sufficient length. Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file, if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

9.2. Method Blank (MB)

9.2.1. One MB must be processed with each preparation batch of up to 20 samples. The MB consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The MB must not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants: see below) or at, or above, 10 % of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of ten times higher than the MB contamination level). For Ohio VAP projects, all analytes must be less than the reporting limit with the following exceptions: (a) insufficient sample for re-digestion (b) expired holding times, or (c) the elements detected in the MB are non-detect for the associated samples. In cases where the analyte is a common laboratory contaminant (copper, iron, lead, or zinc), the data may be reported with qualifiers if the concentration of the analyte in the MB is less than two times the RL. Such action must be addressed in the project narrative.

9.2.1.1. Re-preparation and re-analysis of all samples associated with an unacceptable MB is required when reportable concentrations are determined in the samples (see exception noted above).

9.2.1.2. If there is no analyte greater than the RL in the samples associated with an unacceptable MB, the data may be reported with qualifiers. Such action must be addressed in the project narrative.

9.2.1.3. If the above criteria are not met and re-analysis is not possible, then the sample data must be qualified. This anomaly must be addressed in the project narrative.

9.3. Laboratory Control Sample (LCS)

9.3.1. One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. Aqueous LCS spike levels are provided in Table II (Appendix A). The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.1.1. If any analyte is outside established control limits, the system is out of control and corrective action must occur. Unless in-house control limits are established, a control limit of 80 - 120% recovery must be applied for Method 6010B and 6010C. For Method 200.7, control limits of 85-115% must be applied.

9.3.1.2. In the instance where the LCS recovery is greater than the upper control limit and the sample results are < RL, the data may be

reported with qualifiers. Such action must be addressed in the report narrative.

9.3.1.3. Corrective action will be re-preparation and re-analysis of the batch unless the client agrees that other corrective action is acceptable. For Ohio VAP projects, the LCS must meet acceptance criteria. The laboratory may re-analyze an aliquot of the LCS to verify the outlier; however, if the LCS exhibits the same anomaly upon re-analysis, the sample batch must be re-digested or re-extracted and re-analyzed. The exceptions are as follows: (a) insufficient sample for re-extraction/re-digestion (b) expired holding times, or (c) the LCS is biased high and the samples are non-detect for those analytes.

9.4 Additional information on QC samples can be found in QA Policy QA-003. Ohio VAP projects must reference this SOP instead of policy QA-003 for information on QC samples.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.5.1. One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of, or in addition to, MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table II (Appendix A).

9.5.1.1. If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. For Methods 6010B and 6010C, control limits of 75-125% (70-130% for Method 200.7) recovery and 20% RPD or historical acceptance criteria must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include re-preparation and re-analysis of the sample and MS/MSD. MS/MSD results, which fall outside the control limits, must be addressed in the narrative.

9.5.1.2. If the native analyte concentration in the MS/MSD exceeds four times the spike level for that analyte, the recovery data is reported with a "4" flag.

9.5.1.3. For Method 6010C samples – If the MS/MSD recoveries are unacceptable, the same sample from which the MS/MSD aliquots were prepared should also be spiked with a post digestion spike. Otherwise, another sample from the same preparation batch should be used as an alternative. An analyte spike is added to a portion of a prepared sample, or its dilution, and must be recovered to within 80% to 120% of the known value. If this spike fails, then the dilution test must be run on this sample. If both the MS/MSD and the post digestion spike fail, then matrix effects are confirmed.

9.6 Dilution test

9.6.1 A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per preparation batch must be processed as a dilution test. The test is performed by running a sample at a 5X dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted sample after correction for dilution must agree within 10% of the original sample determination when the

original sample concentration is greater than 50 times the MDL. If the results are not within 10%, the possibility of chemical or physical interference exists and the data is flagged.

9.7 Control Limits

9.7.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.7.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMS.

9.8 Method Detection Limits (MDLs) and MDL Checks

9.8.1 MDLs and MDL Checks are established by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.8.2 MDLs are easily accessible via LIMS.

9.9 Nonconformance and Corrective Action

9.9.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action. Deviations are not allowed for Ohio VAP projects.

10. CALIBRATION AND STANDARDIZATION

10.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the instructions in Appendix F.

10.2. Initial Calibration

10.2.1. Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures. Flush the system with the calibration blank between each standard or as the manufacturer recommends. The calibration curve must consist of a minimum of a blank and a standard. Refer to Appendix F for detailed setup and operation protocols. Refer to Instruction Manuals in laboratory. Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument standardization date and time must be included in the raw data

10.3 Initial Calibration Verification (ICV/ICB)

10.3.1 Calibration accuracy is verified by analyzing a second source standard (ICV) immediately after the initial calibration. For analyses conducted under Method 200.7, the ICV result must fall within ±5% of the true value for that

solution with relative standard deviation <3% from replicate (minimum of two) exposures. For Methods 6010B and 6010C, the ICV must fall within ±10% of the true value for that solution. For Method 6010B, the relative standard deviation must be <5% from replicate (minimum of two) exposures. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The calibration blank is prepared with reagent water to the same concentrations of the acids found in the standards. The calibration blank will also be used for all initial (ICB) and continuing calibration blank (CCB) determinations. The ICB result must fall within ± the RL from zero. If either the ICV or ICB fail to meet criteria, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified (see Sections 11.6 through 11.8 for required run sequence).

10.4 Continuing Calibration Verification (CCV/CCB)

10.4.1 Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples and at the end of the sample run. The CCV is to be a mid-range standard made from a dilution of the calibration standard. The CCV for all methods must fall within ±10% of the true value for that solution. For Methods 6010B and 200.7, the relative standard deviation must be <5% from replicate (minimum of two) exposures. For Method 6010C, there is no criterion for RSD from replicate exposures. A CCB is analyzed immediately following each CCV (see Sections 11.6 through 11.8 for required run sequence). The calibration blank is prepared with reagent water to the same concentrations of the acids found in the standards. The calibration blank will also be used for all initial (ICB) and continuing calibration blank (CCB) determinations. The CCB result must fall within ± RL from zero. If the blank is less than 1/10 the concentration of the action level of interest and no sample is within 10% of the action limit, re-analysis and recalibration are not required before continuation of the run. This is not acceptable for Ohio VAP samples. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV or CCB fails, all affected samples must be re-analyzed with valid CCV/CCB pairs (refer to Section 11.7 for an illustration of the appropriate rerun sequence). Exceptions: If CCB > RL, samples < RL can be reported with an NCM. If CCV is outside of criteria on the high side, samples < RL can be reported with an NCM.

10.5 Interference Check Analysis (ICSA/ICSAB)

10.5.1 The validity of the inter-element correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Table IV (Appendix A) for the details of ICSA and ICSAB composition. Custom multi-element ICS solutions must be used. All analytes must be spiked into the ICSAB solution; therefore, if a non-routine analyte is required, then it must be manually spiked into the ICSAB using a certified ultra-high purity single element solution or custom lab-specific mix. If the ICP will display overcorrection as a negative number, then the non-routine elements

can be controlled from the ICSA. Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium, and magnesium must always be included in all ICP runs.

10.5.2 The ICSA and ICSAB solutions must be run at the beginning of the run (see Sections 11.6 or 11.7 for required run sequence).

10.5.3 The ICSAB results for the interferents must fall within 80 - 120% of the true value. If any ICSAB interferent result fails criteria, the analysis must be terminated, the problem corrected, the instrument re-calibrated, and the samples rerun.

10.5.4 ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within ± 2 times the RL from zero. ICSA results for the non-interfering elements with RLs > 10 µg/L must fall within ± 1 times the RL from zero. If the ICSA results for the non-interfering elements do not fall within ± two times RL (RL ≤10) or ± 1xRL (RL>10) from zero, the field sample data must be evaluated as follows.

10.5.4.1. If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.

10.5.4.2. If the affected element was not required, then the sample data can be accepted.

10.5.4.3. If the interfering elements are not present in the field sample at a concentration which would result in a false positive or negative result greater than ± two times the RL from zero, then the field sample data can be accepted.

10.5.4.4. If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than ± two times the RL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than ten times the analyte signal in the ICSA.

10.5.4.5. If the data does not meet the above conditions, then the IECs must be re-evaluated and corrected if necessary and the affected samples re-analyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually, the calculations must be clearly documented on the raw data.

10.6 CRI (CRA or LLICV/LLCCV(6010C)

10.6.1 To verify linearity near the RL for ICP analysis, a CRI standard is run at the beginning of each sample analysis run. Additionally, some projects may require CRI analysis at the end of the run (see Sections 11.6 or 11.7 for required run sequence). Evaluate associated samples based upon advisory limits of ± 50% of true value. For Method 6010C, it must be analyzed at the beginning and end of the analytical run. The control limit for this method is 70-130%.

Note: The custom CRI mix contains most analytes at a level near the standard lab reporting limit.

11. PROCEDURE

11.1. A minimum of two exposures for each standard, field sample and QC sample is required. The average of the exposures is reported. For Trace ICP analyses, the results of the sum channel must be used for reporting.

11.2. Prior to calibration and between each sample/standard, the system is rinsed with the calibration blank solution.

11.3. The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV, CCV), blanks (ICB, CCB, PB), interference checks (ICSA, ICSAB), and field samples (linear range) to improve the data review process.

11.4. To facilitate the early identification of QC failures and samples requiring rerun, it is strongly recommended that sample data be reviewed periodically throughout the run.

11.5. The following procedural guidelines must be followed when using an internal standard:

11.5.1 Typically used internal standard is Yttrium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)

11.5.2 The internal standard (IS) must be added to every sample and standard at the same concentration. It is recommended that the IS be added to each analytical sample automatically through use of a third pump channel and mixing coil. Internal standards must be added to blanks, samples, and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

11.5.3 The concentration of the internal standard must be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

11.5.4 The internal standard raw intensity counts must be printed on the raw data.

11.5.5 The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte). The instrument automatically adjusts sample results based on comparison of the internal standard intensity in the sample to the internal standard intensity at calibration.

11.5.5.1 If the internal standard counts fall within ±50% of the counts observed in the ICB or calibration blank then the data is acceptable.

11.5.5.2 If the internal standard counts in the field samples are more than ± 50% higher than the expected level, a dilution is needed due to matrix interference.

11.6 The following analytical sequence must be used for Methods 6010B, 6010C, and 200.7:

Instrument Calibration
ICV
ICB
CRI/LLICV (6010C)
ICSA
ICSAB
CCV
CCB
10 samples
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete the run
CRI (The CRI counts as a sample analysis.)/LLICV (6010C)
CCV
CCB

Refer to Quality Control Section 9.0 for Methods 6010B, 6010C, and 200.7 quality control criteria.

11.7 The following run sequence provides an illustration of a mid-run CCV or CCB failure and the appropriate corrective action run sequence as described in Section 10.5.

Original Run: Instrument Calibration
ICV
ICB
CRI
ICSA
ICSAB
CCV

CCB
10 samples
CCV
CCB
10 samples
CCV
CCB
10 samples **
CCV *
CCB *
10 samples **
CCV
CCB
10 samples
CCV
CCB

* Failure occurs at CCV/CCB
**Samples requiring rerun for affected analytes

11.8 The instrument may be re-profiled between CCV/CCB pairs to correct for environment-induced drift.

11.9 Guidelines are provided in the Appendix C, D, and E on procedures to minimize contamination of samples and standards, preventive maintenance, and troubleshooting.

11.10 All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed the linear range. If an inter-element correction exists for an analyte, which exceeds the linear range, the IEC may be inaccurately applied. Therefore, even if an over-range analyte may not be required to be reported for a sample, if that analyte is an interferent for any requested analyte in that sample, the sample must be diluted. Acid strength must be maintained in the dilution of samples.

11.11 Nonconformance documentation must be filed in the project file.

11.12. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.13. Analytical Documentation

11.13.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.13.2. Record all standards and reagents in the LIMS Reagents module. All standards and reagents are assigned a unique number for identification.

11.13.3. Documentation, such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs, is available for each data file.

11.13.4. Record all sample results and associated QC into LIMS. Level I and Level II reviews are performed in LIMS.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%\ R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates is calculated according to the following equation:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration (mg/L) from instrument readout (mean of two exposures)
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The final concentration determined in digested solid samples is calculated as follows:

$$mg / Kg, dry\ weight = \frac{C \times V \times D}{W}$$

Where:
C = Concentration (mg/L) from instrument readout (mean of two exposures)
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:
I = Sample result (Instrument reading)
S = Dilution test result (Instrument reading × 5)

12.9. Appropriate factors must be applied to sample values if dilutions are performed.

12.10. Trivalent Chromium

12.10.1 Trivalent chromium (CR^{+3}) is the result obtained by subtracting the hexavalent chromium (CR^{+6}) results for a sample from the total chromium result from that sample. The total chromium result is determined using the procedures in this SOP. The hexavalent chromium result is determined using the procedures in TestAmerica Canton SOP NC-WC-024.

12.10.2 Reporting Limits

12.10.1 The TestAmerica Canton water reporting limit for trivalent chromium is 0.02 mg/l.

12.10.2 The TestAmerica Canton solid reporting limit for trivalent chromium is 2.0 mg/kg, wet weight.

12.10.3 Calculations: Cr^{+3} = Cr, total – Cr^{+6}

12.10.3 Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points,
CA-T-P-002

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Refer to Table I in Appendix A for the list of analytes that may be analyzed using this SOP.

13.3. Training Qualification

13.3.1 The Group/Team Leader or the Supervisor has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the corporate environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste must be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by this Method

15.2.1. The following waste streams are produced when this method is carried out:

15.2.2. Acid waste consisting of sample and rinse solution: Any sample waste

generated must be collected and disposed of in the acid waste drum located in the Metals Lab.

15.2.3. Standards must be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

16. REFERENCES

16.1. References

16.1.1. 40 CFR Part 136, Appendix B, 7-5-95, Determination of Method Detection Limits

16.1.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010B

16.1.3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Final Update IV, Method 6010C, Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 3, February 2007

16.1.4. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, May 1994. Method 200.7

16.1.5. Inductively Coupled Plasma – Atomic Emission Spectrometric Method for Trace Element Analysis of water and wastes Method 200.7, 40 CFR – Chapter I – Part 136 – Appendix C. Electronic version dated September 30, 2002

16.1.6. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.7. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.8. Corporate Quality Management Plan (CQMP), current version

16.1.9. Revision History

Historical File:		Revision 2.0: 10/27/97		Revision 0: 01/08/04 (NC-MT-012)
(formerly CORP-MT-0001NC)		Revision 2.1: 04/19/99		Revision 1: 01/07/09
		Revision 3.1: 10/04/00		Revision 2: 02/22/11
		Revision 3.2: 01/19/01		
		Revision 3.3: 12/05/01		

		Revision 3.4: 01/08/04		

16.2. Associated SOPs and Policies, current version

16.2.1. TestAmerica Canton QC Program, QA-003

16.2.2. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.3. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.4. Supplemental Practices for DoD Project Work, NC-QA-016

16.2.5. Hexavalent Chromium (Colorimetric), NC-WC-024

16.2.6. Acid Digestion of Soils, SW846 Method 3050B, NC-IP-010

16.2.7. Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods, NC-IP-011

16.2.8. Standards and Reagents, NC-QA-017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference method

17.1.1. Modifications/interpretations from Methods 6010B and 200.7

17.1.1.1. TestAmerica Canton Laboratories use mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in-house as recommended by the subject methods.

17.1.1.2. Methods 200.7 and 6010B state that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution must fall within a specific concentration range around the calibration blank. In determining IECs because of lack of definition clarification for "concentration range around the calibration blank," TestAmerica Canton has adopted the procedure in EPA CLP ILMO4.0.

17.1.1.3. Whenever a new or unusual matrix is encountered, a series of tests be performed prior to reporting concentration data for that analyte. The dilution test helps determine if a chemical or physical interference exists. Because TestAmerica Canton laboratories receive no prior information from clients regarding when to expect a

new or unusual matrix, TestAmerica Canton may select to perform a dilution test on one sample in each prep batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. At TestAmerica Canton, matrix interference is determined by evaluating data for the LCS and MS/MSD. TestAmerica Canton REQUIRES documented, clear guidance when a new or unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

17.1.2. Modifications from Method 200.7

17.1.2.1. Method 200.7 defines the IDL as the concentration equivalent to a signal due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the same wavelength. TestAmerica Canton lab utilizes the IDL definition as defined in Section 9.1 of this SOP.

17.1.2.2. The calibration blank is prepared in an acid matrix of 5% HNO_3/5% HCl instead of the specified 2% HNO_3/10% HCl matrix as the former matrix provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.

17.1.2.3. Method Section 9.3.4 specifies that "Analysis of the ICV (ICSA/AB) solution immediately following calibration must verify that the instrument is within ± 5% of calibration with a relative standard deviation <3% from replicate integrations ≥ 4". TestAmerica Canton uses a minimum of two exposures.

17.1.2.4. The 40 CFR version of Method 200.7 requires the instrument check standard to agree within ± 5% of expected values and less than, or equal to, 3% RSD. Also, the 40 CFR requires the interference check sample to be analyzed at the beginning, end, and at periodic intervals throughout the sample run. At TestAmerica Canton, the instrument check standard equals the CCV, which must agree within ± 10% of expected values and 5% RSD, and the ICSA standards are analyzed only at the beginning of a sample run. TestAmerica's procedures are in line with the Rev. 4.4, May 1994 version of Method 200.7.

17.1.2.5. Section 7.12 of Method 200.7 indicates that the QCS (ICV) must be prepared at a concentration near 1 ppm. The ICV specified in this SOP accommodates the 1 ppm criteria for the majority of analytes. For the remaining analytes, this SOP specifies ICV concentrations which are appropriate to the range of calibration. The intent of the ICV, verification of calibration standard accuracy, is independent of the ICV concentration used.

17.1.2.6. The ICS criteria applied by this SOP differ from those stated in the method. Method 200.7 Section 10.4 states that results must fall within the established control limits of 3 times the standard deviation of the calibration blank for that analyte. The control limits listed in this SOP are those applicable to the EPA designed solution.

17.1.2.7. Method 200.7 Section 9.3.4 states the CCB must be less than the IDL, but more than the lower 3-sigma control limit of the calibration blank. The intent of this requirement is to ensure that the calibration is not drifting at the low end. TestAmerica Canton has adopted an absolute control limit of ± RL from zero for calibration blank criteria. SOP Section 10.5 provides the detailed corrective action criteria that must be followed.

17.1.3. Modifications from Method 6010B

17.1.3.1. Chapter 1 of SW-846 states that the method blank must not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client. This is not acceptable for Ohio VAP projects.

17.1.3.2. Method 6010B Section 8.6.1.3 states that the results of the calibration blank are to agree within three times the IDL. If not, repeat the analysis two or more times, and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, recalibrate, and re-analyze the previous ten samples. The intent of this requirement is to ensure that the calibration is not drifting at the low end. TestAmerica Canton has adopted an absolute control limit of ± RL from zero for calibration blank criteria. See SOP Section 10.5 for a detailed description of the required corrective action procedures.

APPENDIX A - TABLES

TABLE I: Methods 200.7, 6010B, and 6010C Target Analyte List

Element	Symbol	CAS #
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Boron	B	7440-42-8
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Lithium	Li	7439-93-2
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silicon	Si	7440-21-3
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Strontium	Sr	7440-24-6
Thallium	Tl	7440-28-0
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6
Tin	Sn	7440-31-5
Titanium	Ti	7440-32-6

TABLE II: Matrix Spike and Aqueous Laboratory Control Sample Levels

Element	Concentration (ug/L)
Aluminum	2000
Antimony	500
Arsenic	2000
Barium	2000
Beryllium	50
Cadmium	50
Calcium	50000
Chromium	200
Cobalt	500
Copper	250
Iron	1000
Lead	500
Lithium	1000
Magnesium	50000
Manganese	500
Molybdenum	1000
Nickel	500
Potassium	50000
Selenium	2000
Silicon	1000
Silver	50
Sodium	50000
Strontium	1000
Thallium	2000
Vanadium	500
Zinc	500
Boron	1000
Tin	2000
Titanium	1000

TABLE III: Trace ICP Calibration and Calibration Verification Standards

Element	Calibration Level	ICV (ug/L)	CCV (ug/L)
Aluminum	50000	12500	25000
Antimony	1000	250	500
Arsenic	1000	250	500
Barium	4000	1000	2000
Beryllium	4000	1000	2000
Cadmium	1000	250	500
Calcium	100000	25000	50000
Chromium	4000	1000	2000
Cobalt	4000	1000	2000
Copper	4000	1000	2000
Iron	50000	12500	25000
Lead	1000	250	500
Lithium	10000	1000	5000
Magnesium	100000	25000	50000
Manganese	4000	1000	2000
Molybdenum	4000	1000	2000
Nickel	4000	1000	2000
Potassium	100000	25000	50000
Selenium	1000	250	500
Silicon	10000	3000	5000
Silver	2000	500	1000
Sodium	100000	25000	50000
Strontium	10000	4500	5000
Thallium	2000	500	1000
Vanadium	4000	1000	2000
Zinc	4000	1000	2000
Boron	10000	1000	5000
Tin	10000	1000	5000
Titanium	10000	1000	5000

TABLE IV: Interference Check Sample Concentrations

Element	ICSA (ug/L)	ICSAB (ug/L)
Aluminum	500000	500000
Antimony	-	1000
Arsenic	-	1000
Barium	-	500
Beryllium	-	500
Cadmium	-	1000
Calcium	500000	500000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200000	200000
Lead	-	1000
Lithium		500
Magnesium	500000	500000
Manganese	-	500
Molybdenum	-	1000
Nickel	-	1000
Potassium	-	10000
Selenium	-	1000
silicon		10000
Silver	-	1000
Sodium	-	10000
Strontium		1500
Thallium	-	1000
Vanadium	-	500
Zinc	-	1000
Tin	-	1000
Boron		1000
Titanium		1000

APPENDIX B - CROSS REFERENCE OF TERMS COMMONLY USED IN METHODS EPA 200.7, SW 6010B, 6010C, AND TESTAMERICA CANTON SOP

EPA 200.7	SW 6010B / 6010C	TestAmerica Canton SOP
Calibration blank (CB)	Calibration blank	Initial and continuing calibration blanks (ICB/CCB)
Dilution test	Dilution test	Dilution Test
Instrument detection limit (IDL)	Instrument detection limit (IDL)	Instrument detection limit (IDL)
Instrument performance check (IPC)	Continuing calibration verification (CCV)	Continuing calibration verification (CCV)
Internal standard	Internal standard	Internal standard (IS)
Laboratory duplicates	N/A	N/A
Laboratory fortified blank (LFB)	N/A	Laboratory control sample (LCS)
Laboratory fortified sample matrix (LFM)	Matrix spike and matrix spike duplicate (MS/MSD)	Matrix spike and matrix spike duplicate (MS/MSD)
Laboratory reagent blank (LRB)	Method blank	Method or Prep blank (MB)
Linear dynamic range (LDR)	Linear dynamic range (LDR)	Linear dynamic range (LDR)
Method detection limit (MDL)	Method detection limit (MDL)	Method detection limit (MDL)
Quality control sample (QCS)	Check standard or Initial calibration verification (ICV)	Initial calibration verification (ICV)
Spectral interference check solution (SIC)	Interference check solution (ICS)	Interference check solution (ICSA/ICSAB)

APPENDIX C - TROUBLESHOOTING GUIDE

Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer Clean or replace mixing chamber
Instrument Drift	Replace torch (Crack) Clean or replace nebulizer (blockage) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Re-profile
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage (back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

APPENDIX D - CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware must be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the Metals Lab. All work areas must be kept scrupulously clean.

Powdered Gloves must not be used in the Metals Lab since the powder contains silica and zinc as well as other metallic analytes. Glassware must be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipette tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

APPENDIX E - PREVENTATIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs, indicate the date, time and instrument number. Then identify the problem and corrective action in the Maintenance Log.

The following procedures are required to ensure that that the instrument is fully operational:

Change sample pump tubing and pump windings

As Needed:

- Check rinse solution and fill if needed
- Check waste containers and empty if needed
- Check sample capillary tubing is clean and in good condition
- Check droplet size to verify nebulizer is not clogged.
- Check sample flow for cross flow nebulizer
- Check pressure for vacuum systems
- Clean plasma torch assembly to remove accumulated deposits
- Clean nebulizer and drain chamber; keep free-flowing to maintain optimum performance
- Replace peristaltic pump tubing, sample capillary tubing and autosampler sipper probe
- Apply silicon spray on autosampler tracks
- Check water level in cool flow
- Change oil for vacuum systems
- Replace coolant water filter (may require more or less frequently depending on quality of cooling water)

APPENDIX F - ICP Operating Instructions
ICP Analysis (TJA 61E) Example

1. SETUP

a. Plasma Control Panel (enter)
b. (F1) - Startup
c. (F9) - Continue
d. (F2) - Levels
    1. Change auxiliary gas to low – use space bar to toggle
    2. Change nebulizer gas flow to 0.5 L/min.
    3. Change pump rate to 130
    4. Escape
    5. Allow instrument to warm up approximately 30 minutes.

2. DEVELOPMENT

a. Methods (enter)
b. Enter method name
c. (F3)-Method Info.
d. Change file name
e. (F9) - Done
f. (F9) - Done/Keep

3. OPERATION

a. Analysis (enter)
b. (F5)-Profile
    1. (F3) - Automatic
    2. (F1) - Run
    3. If peak position is greater than ± 0.05 units from the center peak position, you must adjust the profile. If it is within ± 0.05 units, press (F9) - Done.
    4. To adjust select (F1) - CalcSS and enter current vernier position. (enter)
    5. Adjust to new vernier position (F9) - Done
    6. Rerun profile until peak position is ± 0.05 units.
    7. (F9) - Done
c. Autosampler (F9)
    1. Enter method name (enter)
    2. Enter autosampler table name (enter)
    3. (F1) - Run

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Canton

SOP No. NC-MT-014, Rev. 3
Effective Date: 06/05/13

Title: PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS AND SOLID SAMPLES BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY

[Method: MCAWW Method 245.1, SW846 Method 7470A, SW846 7471A, and 7471B]

Approvals (Signature/Date):

Susan M. Girard	05/23/13	[redacted]	05/29/13
Technology Specialist	Date	Health & Safety Coordinator	Date
Lorenz Strait	05/23/13	[redacted]	05/24/13
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. NC-MT-014, Rev 2, dated 03/20/13

Facility Distribution No. _____ **Distributed To: _____**

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW846 Method 7470A, MCAWW Method 245.1 Method 7471B, and Method 7471A.

1.2. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants and potassium permanganate has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity, and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation, and volume of sample used.

1.3. Method 7470A is applicable to the preparation and analysis of mercury in ground water, aqueous samples, TCLP, and other leachates/extracts. Certain solid and sludge type wastes may also be analyzed; however, Method 7471A is usually the method of choice. All matrices require sample preparation prior to analysis.

1.4. Method 245.1 is applicable to the determination of mercury in drinking, surface and saline waters, and domestic and industrial wastes. All matrices require sample preparation prior to analysis.

1.5. Methods 7471A and 7471B are applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits, wastes, wipes, biological material, and sludge-type materials. All matrices require sample preparation prior to analysis.

1.6. The TestAmerica North Canton reporting limit for mercury in aqueous matrices is 0.0002 mg/L except for TCLP or SPLP leachates for which the reporting limit is 0.002 mg/L. The TestAmerica North Canton reporting limit for mercury in solid matrices is 0.1 mg/kg.

1.7. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric (aqueous samples), or hydrochloric and nitric acids (soil samples). Organic mercury compounds are oxidized with potassium permanganate (aqueous and soil samples) and potassium persulfate (aqueous samples), and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current version or Table 2: Glossary of Acronyms located at the end of this document.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control (QC) section. All glassware is cleaned per SOP NC-QA-014. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Chemical and physical interferences may be encountered when analyzing samples using this method.

4.3. Potassium permanganate, which is used to break down organic mercury compounds, also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.4. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.5. Chlorides can cause a positive interference. Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (maximum 25 mL); because during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride

4.6. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.7. Samples containing high concentrations of oxidizable organic materials, as evidenced by high Chemical Oxygen Demand (COD) levels, may not be completely oxidized by this procedure. When this occurs, the recovery of mercury will be low. Reducing the volume of original sample used can eliminate this problem.

4.8. The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. Refer to Appendix B for Contamination Control Guidelines.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Material Safety Data Sheet (MSDS) for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit(2)	Signs and symptoms of exposure
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 g/m^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns

			and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydroxyl-amine Hydro-chloride	Corrosive Poison	None	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Corrosive to the eyes. Irritant and possible sensitizer. May cause burns to the skin.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
Potassium Permanganate	Oxidizer	5 mg/m^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.5 Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.7 Do not look directly into the beam of the Hg lamp. The Ultra Violet (UV) light that

these lamps radiate is harmful to the eyes.

5.8 Cylinders of compressed gas must be handled with caution in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory, and the gas led to the instrument through approved lines.

5.9 The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6 EQUIPMENT AND SUPPLIES

6.1 Temperature-controlled hot block or equivalent (henceforth referred to as Hot Block).

6.2 Atomic Absorption Spectrophotometer equipped with:

6.2.1 Absorption cell with quartz end windows perpendicular to the longitudinal axis: Dimensions of the cell must result in sufficient sensitivity to meet the SOP defined reporting limit. The quartz windows must be maintained to provide accurate measurements. Any scratches or fingerprints can alter the absorption of UV radiation.

6.2.2 Mercury-specific hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL)

6.2.3 Peristaltic pump which can deliver 1 L/min air

6.2.4 Flowmeter capable of measuring an airflow of 1 L/min

6.2.5 Recorder or printer

6.2.6 Drying device to prevent condensation in cell.

Note: Instruments designed specifically for the measurement of mercury using the cold vapor technique may be substituted for the atomic absorption spectrophotometer.

6.3 Plastic bottles – capable of holding 100 mL

6.4 Nitrogen or argon gas supply, welding grade or equivalent

6.5 Calibrated automatic pipettes

6.6 Class A volumetric flasks

6.7 Top-loading balance, capable of reading up to two decimal places

6.8 Thermometer (capable of accurate readings at 95 °C)

6.9 Disposable cups or tubes

7 REAGENTS AND STANDARDS

7.1 Reagent water must be produced by a Millipore Deionized Water (DI) system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2 Stock (10 ppm calibration and 1 ppm ICV) mercury standards are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Additional information can be found in SOP NC-QA-017. Refer to the reagent module in the Laboratory Information Management System (LIMS) for details on standard preparation.

7.3 Working mercury standard (0.1 ppm): Take 1 mL of the 10 ppm stock mercury standard (Section 7.2) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO_3. This acid (150 uL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot. Refer to the reagent module in LIMS for details on standard preparation.

7.4 The calibration standards must be prepared fresh daily from the working standard (Section 7.3) by transferring 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working mercury standard and 0.5 mL of the stock mercury standard into sample preparation bottles for solid samples and/or by transferring 0, 0.1, 0.25, 0.5, 2.5, and 5.0 mL aliquots of the working mercury standard and 0.25 mL of the stock mercury standard into sample preparation bottles for aqueous samples and proceeding as specified in Section 11.1. The laboratory control sample (LCS) solution is prepared by transferring 5.0 mL of working standard (Section 7.3) into sample preparation bottles and proceeding as specified in Section 11.1. Refer to the reagent module in LIMS for details on standard preparation.

Note: Alternate approaches to standard preparation may be taken, and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I (Appendix A) are maintained. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.5 The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.

7.6 Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed through the entire analytical procedure including sample preparation.

7.7 Nitric acid (HNO_3), concentrated, trace metal grade or better

7.8 Hydrochloric acid (HCl), concentrated, trace metal grade or better

7.9 Sulfuric acid (H_2SO_4), concentrated, traces metal grade or better.

7.10 Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

7.11 Stannous chloride solution: Add 50g ± 0.5g of stannous chloride and 25 mL of concentrated HCl, and bring to a final volume of 500 mL with DI water.

Note: Stannous sulfate may be used in place of stannous chloride. Prepare the stannous sulfate solution according to the recommendations provided by the instrument manufacturer.

7.12 Sodium chloride-hydroxylamine hydrochloride solution: Add 240g ± 0.5g of sodium chloride and 240g ± 0.5g of hydroxylamine hydrochloride to every 2000 mL of reagent water.

7.13 Potassium permanganate, 5% solution (w/v): Dissolve 100g of potassium permanganate for every 2000 mL of reagent water.

7.14 Potassium persulfate, 5% solution (w/v): Dissolve 100 g of potassium persulfate for every 2000 mL of reagent water.

8 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample holding time for mercury is 28 days from time of sample collection to the time of sample analysis.

8.2 Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3 Soil samples and biological material do not require preservation, but must be collected in wide-mouth glass jars with PFTE-lined lids and stored at 4° C ± 2° C (and/or freezing for tissues) until the time of analysis.

9 QUALITY CONTROL

9.1 Initial Demonstration of Capability

9.2 Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well-characterized, laboratory-generated sample used to monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.2.1 Four aliquots of the laboratory check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.3 Preparation Batch - A group of up to 20 samples, excluding QC Samples (Laboratory Control Sample (LCS), Method Blank (MB), Matrix Spike (MS), Matrix Spike Duplicate (MSD)), that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain an MB, an LCS and an MS/MSD. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes. In some cases, at client request, it may be appropriate to process a MS and sample duplicate (DU) in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.4 Method Blank (MB) - One MB must be processed with each preparation batch. The MB consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The MB must not contain any analyte of interest at, or above, the reporting or at, or above, 10% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of ten times higher than the MB contamination level).

Note: For Ohio VAP projects, the result must be below the reporting limit or samples must be re-digested and re-analyzed unless the samples are non-detect.

- Re-digestion and re-analysis of all samples associated with an unacceptable MB is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable MB, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

- If the above criteria are not met and re-analysis is not possible due to limited sample quantity, then the sample data must be qualified. **This anomaly must be addressed in the project narrative.**

9.5 Laboratory Control Sample (LCS) – One LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The LCS must be carried through the entire analytical procedure. If the LCS is outside established control limits, the system is out of control and corrective action must occur. See Section 12 for the LCS calculation.

- In the instance where the LCS recovery is greater than the upper control limit and the sample results are less than RL, the data may be reported. Such

action must be addressed in the project narrative. For Method 245.1, the LCS must be 85% - 115%. For Method 7471B, the laboratory control sample recovery must be 80%-120%.

- Corrective action must be re-digestion and re-analysis of the batch unless the client agrees that other corrective action is acceptable. For Ohio VAP projects the corrective action must be re-digestion and reanalysis of the batch.

9.6 Additional information on QC samples can be found in QA Policy QA-003. Ohio VAP projects must reference this SOP instead of policy QA-003 for information on QC samples.

9.7 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. An MS is a field sample to which known concentrations of target analytes have been added. An MSD is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and MS. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates (DU) in place of, or in addition to, MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table I (Appendix I). See Section 12 for the matrix spike/matrix spike duplicate (MS/MSD) and Relative Percent Difference (RPD) calculation.

- If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. A control limit of=, 70 – 130% for Method 245.1, and 20% RPD must be applied to the MS/MSD. A control limit of 80-120% for Method 7471B and 20% RPD must be applied to the MS/MSD. If the LCS recovery is within control limits, then the laboratory operation is in control and the results may be accepted. Client specific MS/MSD samples may require corrective action. Such action must be addressed in the project narrative by means of a non-conformance memo (NCM). If the recovery of the LCS is outside of control limits, corrective action must be taken. Corrective action must include re-digestion and re-analysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds four times the spike level for that analyte, the recovery data are reported with a "4" as a flag. In the event an MS/MSD analysis is not possible, notation in the project narrative is required.

9.8 Control Limits

9.8.1 Control limits are established by the laboratory as described in SOP NC-QA-018

9.8.2 Control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMS

9.9 Method Detection Limits (MDLs) and MDL Checks

9.9.1 MDLs and MDL Checks are established by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.9.2 MDLs are easily accessible via LIMS

9.10 Nonconformance and Corrective Action

9.10.1 Any deviations from QC procedures must be documented as a nonconformance. Procedural deviations are not allowed for Ohio VAP Projects.

10 CALIBRATION AND STANDARDIZATION

10.1 Calibration standards must be processed through the preparation procedure as described in Section 11.1.

10.2 Due to the differences in preparation protocols, separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.3 Calibration must be performed daily and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.4 Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the CVAA instrument manual for detailed setup and operation protocols.

10.5 Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a calibration blank. One standard must be at, or below, the reporting limit. Analyze standards in ascending order beginning with the calibration blank. Refer to Section 7 and Table I for additional information on preparing calibration standards and calibration levels.

10.6 The calibration curve must have a correlation coefficient of ≥ 0.995, or the instrument must be stopped and recalibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient. NOTE: If any digested calibration standard does not meet SW846 criteria, all associated Ohio VAP samples must be re-digested.

10.7 Initial Calibration Verification/Initial Calibration Blank (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard ICV. The ICV result must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall

within ± the reporting limit (RL) from zero. See Section 12 for the ICV calculation. If either the ICV or ICB fail to meet criteria, the analysis must be terminated, the problem corrected, and the instrument recalibrated (see Section 11.2.6 for required run sequence). The calibration curve standards are reanalyzed to determine if the failure was instrument related. If the cause of the ICV or ICB failure was not directly instrument-related, the corrective action must include re-digestion of the ICV, ICB, CRA, CCV, and CCB with the calibration curve. For Ohio VAP, the sample batch must be re-digested.

10.8 Continuing Calibration Verification/Continuing Calibration Blank (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV must be a mid-range standard at a concentration other than that of the ICV.

10.8.1 For Method 245.1, the CCV must be 5% immediately following the calibration. All other CCVs for Method 245.1 must be 90-110%.

10.8.2 The CCV result for Methods 7470A, 7471A, and 7471B must fall within 20% of the true value for that solution. See Section 12 for the CCV calculation.

10.8.3 A CCB is analyzed immediately following each CCV (see Section 11.2.6 for required run sequence). The CCB result must fall within ± RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. If the CCV/CCB is biased high and the sample results associated with the CCV/CCB are below the requested reporting limit, then the results can be reported. Sample results may be reported when bracketed by valid CCV/CCB pairs. If any digested calibration standard does not meet SW846 criteria, all associated Ohio VAP samples must be re-digested.

10.9 Detection Limit Standard (CRA) - To verify linearity at the reporting limit, a CRA standard is run at the beginning of each sample analysis run after the ICV/ICB. The CRA standard mercury concentration is 0.2 ug/L. It is recommended that the recovery be ± 50% of the true value, or the standard is either rerun or the problem corrected and the instrument re-calibrated. The CRA is only required when requested.

10.10 For DoD work, refer to SOP NC-QA-016 for specific details.

11 PROCEDURE

11.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described. Procedural deviations are not allowed for Ohio VAP projects.

11.3 Standard and Sample Preparation-Solids

11.3.1 All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples.

11.3.2 Transfer 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working standard and 0.5 mL of the stock standard (Section 7.3) into a series of sample digestion bottles. The ICB/CCB consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. For the ICV, transfer a 0.5 ml aliquot of the stock standard. The ICV stock standard must be from a source other than that used for the calibration standards. For the CCV, transfer a 10 mL aliquot of the working standard into a sample digestion bottle.

Note: Alternate volumes and concentrations of standard may be prepared as long as the accuracy and final standard concentrations support laboratory or project reporting limits.

11.3.3 Add reagent water to each standard bottle to make a total volume of 10 mL. Continue preparation as described under Section 11.2 below.

11.3.4 Transfer 0.6g of a well-mixed sample into a clean sample digestion bottle. Continue preparation as described under Section 11.2.

11.4 Hot Block Protocol – Solid Samples

11.4.1 To each LCS **standard** bottle, add 5 mL of reagent water, 5 mL of Aqua Regia, and 5 mL of the working mercury standard (0.1 ppm) (see Section 7.3).

11.4.2 To each **sample** and MB bottle, add 10 mL of reagent water and 5 mL of Aqua Regia.

11.4.3 Heat for two minutes in a hot block at 90 - 95 ° C.

11.4.3.1 Add 40 mL of distilled water.

11.4.3.2 Add 15 mL of potassium permanganate solution. Cover containers.

11.4.3.3 Heat for 30 minutes in the hot block at 90 - 95 °C.

11.4.3.4 Cool

11.4.3.5 Add 6 mL of sodium chloride-hydroxylamine hydrochloride solution to reduce the excess permanganate.

11.4.3.6 To each **standard, quality control sample,** and sample bottle, add 30 mL of reagent water.

Continue as described under Section 11.5.

11.5 Standard and Sample Preparation Waters

11.5.1 All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples. Transfer 0, 0.1, 0.25, 0.5, 2.5 and 5.0 mL aliquots of the working standard and 0.25 mL of the stock standard (Section 7.3) into a series of sample digestion bottles. For the ICV, transfer a 0.25 mL aliquot of the stock standard. The ICV stock standard must be from a source other than that used for the calibration standards. For the CCV, transfer a 5 mL aliquot of the working standard into a sample digestion bottle. The Method Blank (MB) consists of 50 mL of reagent water.

Note: Alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations support laboratory or project reporting limits.

11.5.1 Transfer 50 mL of well-mixed sample or standard to a clean sample digestion bottle. Continue preparation as described under Section 11.4.

Note: Reduced sample volumes can be used as long as a representative sample can be obtained and the reagent levels are adjusted to maintain the same sample to reagent ratio. All samples and standards must be processed similarly.

Note: Spiking is done before the addition of acids or reagents.

11.6 Hot Block Protocol – Water Samples

11.6.1 Add 2.5 mL of concentrated H_2SO_4 and 1.25 mL of concentrated HNO_3.

11.6.2 Add 7.5 mL of potassium permanganate solution. For samples high in organic materials or chlorides, additional permanganate may be added. Shake and add additional portions of permanganate solution until a purple color persists for at least 15 minutes. If after the addition of up to 12.5 mL additional permanganate the color does not persist, sample dilution prior to re-analysis may be required.

Note: The sample dilution resultant from the addition of more than the original aliquot of permanganate solution must be compensated for in the final calculation.

11.6.3 Add 4 mL of potassium persulfate solution, cover, and heat for two hours in a hot block at 90 - 95 °C.

11.6.4 Cool samples.

11.6.5 Add 3 mL of sodium chloride-hydroxylamine hydrochloride solution to reduce the excess permanganate.

11.7 Sample Analysis

11.7.1 Automated determination. Refer to Appendix C for instrument setup and operation.

11.7.2 Perform a weighted linear regression analysis of the calibration standards by plotting maximum response of the standards vs. concentration of mercury. Determine the mercury concentration in the samples from the weighted linear regression fit of the calibration curve. The calibration acceptance criteria are listed in Section 10.6. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.

11.7.3 All measurements must fall within the defined calibration range to be valid. Dilute and re-analyze all samples for analytes that exceed the highest calibration standard.

11.7.4 The following analytical sequence is consistent with Methods 7470A, 245.1, 7471A and 7471B.

Instrument Calibration
ICV
ICB
CRA if required
Maximum 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 for quality control criteria.

Note: Samples include the MB, LCS, MS, MSD, duplicate, field samples and sample dilutions.

11.7.5 To facilitate the early identification of QC failures and samples requiring rerun, it is strongly recommended that sample data be reviewed periodically throughout the run.

11.7.6 Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance, and troubleshooting.

11.8 Analytical Documentation

11.8.2 Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.8.3 Record all standards and reagents in the LIMS Reagents module. All standards and reagents are assigned a unique number for identification.

11.8.4 Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.8.5 Record all sample results and associated QC in LIMS. Level I and Level II review is performed in LIMS.

12. DATA ANALYSIS AND CALCULATIONS

12.1 ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2 CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3 Matrix spike (MS) recoveries are calculated according to the following equation:

$$\%\ R\ =\ 100\left(\frac{SSR\ -\ SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4 The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
Matrix Spike (MS) = determined spiked sample concentration
Matrix Spike Duplicate (MSD) = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:
DU1 = Sample result
DU2 = Sample duplicate result

12.5 The final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg,\ dry\ weight = (C \times V \times D)/(W \times S)$$

Where:
C = Concentration (ug/L) from instrument readout
V = Volume of digestate (L)
D = Instrument dilution factor
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor must be omitted from the above equation.

12.6 The final concentration for an aqueous sample is calculated as follows:

$$mg/L = C \times D$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor

12.7 The Laboratory Control Sample (LCS) percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8 Appropriate factors must be applied to sample values if dilutions are performed.

12.9 Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002.

13. METHOD PERFORMANCE

13.1 Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.1.

13.2 Training Qualification

13.2.1 The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14 POLLUTION PREVENTION

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15 WASTE MANAGEMENT

15.1 All waste must be disposed of in accordance with Federal, State, and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method.

and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2 Waste Streams Produced by this Method

15.2.1 The following waste streams are generated by this method.

15.2.1.1 Acid Waste. This waste disposed of in the designated container labeled "Acid Waste".

15.2.1.2 Acid waste-aqueous waste generated by the analysis. Samples are disposed of in the acid waste drum located in the Metals lab. The contents of the drum are neutralized and released to the POTW.

16. REFERENCES

16.1 References

16.1.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury)

16.1.2 "Methods for the Chemical Analysis of Water and Wastes", Rev. 3.0 (1994)

16.1.3 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A

(Mercury)

16.1.4 Test Methods for Evaluating Solid Waste, Physical/ChemicalMethods, SW-846, 3rd Edition, Revision 2, February 2007, Method 7471B (Mercury)

16.1.5 TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.6 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.7 Corporate Quality Management Plan (CQMP), current version

16.1.8 Revision History

Historical File: (formerly Corp-MT-0007NC, NC-MT-011, and NC-MT-013)				
Revision 1.1: 04/17/97		(NC-MT-011) Rev 0: 12/12/07		Revision 2: 03/20/13
Revision 2.2: 02/06/01		(NC-MT-011) Rev 1: 01/07/09		
Revision 2.3: 05/15/01		(NC-MT-013) Rev 0: 01/07/09		
Revision 2.4: 10/28/02		(NC-MT-014) Rev 0: 09/27/10		
Revision 2.5: 11/24/04		Revision 1-A: 04/17/12		

16.2 Associated SOPs and Policies, current version

16.2.1 QA Policy, QA-003

16.2.2 Glassware Washing, NC-QA-014

16.2.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4 Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5 Supplemental Practices for DoD Project Work, NC-QA-016

16.2.6 Standards and Reagents, NC-QA-017

16.2.7 Calibration Curves (General), CA-Q-S-005

16.2.8 Section of Calibration Points, CA-T-P-002

16.2.9 Subsampling, NC-IP-001

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1 Modifications/Interpretations from Reference Method

17.1.1 Modifications from Method 7471A

17.1.1.1 Chapter 1 of SW846 specifies the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.1.2 Chapter 1 of SW-846 states that the method blank must not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.1.2 Modifications from both Methods 7470A and 245.1

17.1.2.1 The 200 series methods and Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.2.2 This SOP allows for the use of reduced sample volumes to decrease waste generation. Reagent levels are adjusted to maintain the same ratios as stated in the source methods. According to a letter from Robert Booth of EPA EMSL-Cinn to David Payne of EPA Region V, "Reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology."

17.1.3 Modifications from Method 7470A

17.1.3.1 Chapter 1 of SW-846 states that the method blank must not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit if the samples associated with the method blank are equal to or above the reporting limit.

17.1.4 Modifications from Method 245.1

17.1.4.1 Method 245.1 states that standards are not heated. TestAmerica North Canton prepares heated standards for this method.

17.1.4.2 Stannous Chloride is prepared in hydrochloric acid, instead of sulfuric acid, per instrument manufacturer recommendations.

17.1.4.3 Section 9.3.4 of the method states that the CCB must be less than the MDL. The laboratory uses the criteria that the CCB result must fall within ± RL from zero.

APPENDIX A - TABLES

TABLE 1. MERCURY REPORTING LIMITS, CALIBRATION STANDARD, QC STANDARD AND SPIKING LEVELS

Soil RL (mg/kg)	0.1
Standard Aqueous RL (mg/L)	0.0002
TCLP RL (mg/L)	0.002
Std 0 (mg/L)	0
Std 1/CRA (mg/L)	0.0002
Std 2 (mg/L)	0.0005
Std 3 (mg/L)	0.001
Std 4 (mg/L)	0.005
Std 5 (mg/L)	0.010
Std 6 (mg/L)	0.050
ICV (mg/L)	0.005
CCV/LaboratoryControl Sample (LCS) (mg/L)	0.010
LCS (mg/L)	0.005
Matrix Spike (MS) (mg/L)	0.001
TCLP Matrix Spike (MS)	0.005

Table 2 GLOSSARY OF ACRONYMS	
CCB	ContinuingCalibrationBlank
CCV	Continuing Calibration Verification
CF	CorrectionFactor
COC	Chain of Custody
CQMP	Corporate Quality Management Plan
DOC	Demonstration of Capability
DOD	Department Of Defense
EH&S	EnvironmentalHealth and Safety
FID	Flame Ionization Detector
GC	Gas Chromatography
ICB	Initial Calibration Blank
ICV	Initial Calibration Verification
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
MB	Method Blank
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheet
NCM	Non ConformanceMemo

Table 2

GLOSSARY OF ACRONYMS

OSHA	Occupational Safety and Health Administration
PEL	Permissible Exposure Limit
PTFE	Polytetrafluoroethylene
QAM	Quality Assurance Manual
QA/QC	Quality Assurance/QualityControl
RF	Response Factor
RPD	Relative Percent Difference
SOP	Standard Operational Procedure
STEL	Short Term Exposure Limit
TWA	Time Weighted Average
UHP	Ultra High Purity
VOA	Volatiles

APPENDIX B - CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by Deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas should be kept scrupulously clean.

Powdered Gloves should not be used in the metals laboratory since the powder contains zinc, as well as other metallic analytes. Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

APPENDIX C – INSTRUMENT SETUP

Hg Analysis (Leeman PS200II)
System Initialization and Warmup

To Set Up Instrument for Analysis

1. F1 Menu
2. **A**utosampler
    A. **R**ack Entry
    B. **E**dit (ex. Rack 1), Enter
    C. Cup **I**D - Enter (clears sample #'s)
    E. Press Insert Key and move cursor with arrows to cup ID and begin typing labels.
3. Press F2 Macro key and type in – Hg
    A. Enter folder name - ex., HG0306, Enter. If folder does not exist, type Y - Enter.
    B. Type in: "Rack 1", "Rack 2" etc., Enter.
    C. Type in: FROM CUP TO CUP
        Ex. = 1 30

Do the same for Position 2 if needed. If not needed, you must press "Enter" three times to begin analysis.

Hg Analysis(HYDRAAA)

Instrument Instruction

H4 Analysis

There are 3 separate screens to use--WinHgRunner, WinHgDatabase, and Rack Editor

(Turn on Hg lamp prior to analysis)

1. To Set your Protocol and Dataset

   WinHgDatabase – Select a previous protocol, then Save Protocol. Type in file name (ex.: HG40101), then select the (RN) key. This will take you to the WinHgRunner screen.

   WinHgRunner – File, New, type in dataset name (ex.: 0101A). Type in batch name (ex.: Water or Solid). Go back to WinHgDatabase to locate the new protocol.

2. Typing Labels

   Rack Editor - File, New (pick 44 rack). Type in labels under sample ID, and Save As (ex.: 0101A).

3. Activate Gas and Pump

   WinHgRunner – under Control tab, turn on Gas and pump, and pour calibration standards.

4. To Calibrate Curve

   WinHgRunner – under Standard tab, select S1 S2 S3 S4 S5 S6 S7 Rep2. To begin analyzing, select Stnd Auto tab.

5. To Check Calibration Curve

   WinHgDatabase – under Cal Curve tab check that the linear range >0.995, select "Wt.Lin." from the "Type" drop-down list and then accept curve.

6. Verification Standards

   WinHgRunner – under the Standard tab, select C1 (ICV) C2 (ICB) C3 (CRA). To begin analysis, select Ck Std Auto tab.

7. Checking Verification Standards:

   WinHgRunner – Select the Report Tab to review results.

8. To Begin Analyzing Sample with CCV and CCB:

   WinHgRunner – under Standard tab, select C4 (CCB) and C5 (CCV).

   WinHgRunner – under Sample tab, type in rack name, start cup, end cup, cups per rack (44). To start analysis, select the Run Auto tab.

9. View Results

   WinHgRunner – Select the Report tab.

PRINTING REPORTS

1. To Print Cal Curve

   WinHgDatabase – under Cal Curve tab, select Print Cal to print curve.

2. To Print Report

   WinHgDatabase – under Report tab, under Format, turn on Report. Then select Generate to print.

3. To Transfer Run

For instrument H1

- Hit F4 to bring up the report menu
- Type hotkey 'K' to select a disk to write the run to, then type "A:/name-of-run"
- Ex/ for the first run on Nov 7th, type "A:/hg11107A"
- Type hotkey 'L' to select the .prn file type
- Type hotkey 'G' to generate the file (i.e. write the run to the floppy disk)

  Walk the floppy disk to instrument H4's computer, locate the run on the disk, right-click it, and select "Send to? TALSImportH1"

For instrument h4

- **WinHgDatabase** – under Report tab under Format, turn on PRN file and type in file (ex. N:/Inorganics/M1109/name-of-run) where "M1109" is the year and month of the current run. For this example "M1109" is Sept. 2011

- Locate the run that was just saved on the N: drive, right-click it, and select "Send to? TALSImportH4"

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

CANTON

SOP No. NC-WC-031, Rev. 9
Effective Date: 03/22/13

Title: CYANIDE AUTOMATED, PYRIDINE-BARBITURIC ACID METHOD

[Method: SW846 Method 9012A, 9012B, EPA Methods 335.1, 335.2, 335.2 (CLP-M), 335.4, and Standard Methods 4500-CN-E, 4500-CN-I, and 4500-CN-G]

Approvals (Signature/Date):

Technology Specialist Date 3/22/2013

Health & Safety Coordinator Date 3/22/2013

Quality Assurance Manager Date 3/22/2013

Laboratory Director Date 3/22/2013

This SOP was previously identified as SOP No. NC-WC-031, Rev 8.4-A, dated 04/16/12

Facility Distribution No. _____ **Distributed To:_____**

TABLE OF CONTENTS

1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Total, Amenable, and Weak Acid Dissociable Inorganic Cyanide in solids, liquids, and waters. It is based on SW846 Method 9012A, 9012B, 335.1, 335.2, 335.2(CLP-M), 335.4 and Standard Methods 4500-CN-E, 4500-CN-I, and 4500-CN-G. The working linear range is 0.005 – 0.2 mg/L for waters and 0.25-10 mg/kg for solids. Reporting limits are listed in Section 17.

1.2. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. The Cyanide, as HCN, is released by distilling/refluxing the sample with strong acid and is trapped in a sodium hydroxide solution.

2.2. The sodium hydroxide solution is analyzed colorimetrically on an autoanalyzer using the pyridine-barbituric acid method.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current version. For a glossary of acronyms, see Appendix I at the end of this document.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. All glassware is cleaned per SOP NC-QA-014. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Sulfides interfere, but can be eliminated by treating the sample with cadmium carbonate prior to analysis.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS**

for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Cyanide	Poison Corrosive	5 mg/m^3 TWA as CN	**This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heart beat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3 2 mg/m^3-Ceiling	This material will cause burns if comes into contact with the skin or eyes. Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Potassium Phosphate	Flammable	None	Inhalation causes severe irritation to the respiratory tract. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. .
Sodium Phosphate		None	Inhalation may cause respiratory tract irritation. Can produce delayed pulmonary edema. Causes mild skin and eye irritation. Ingestion may cause gastrointestinal irritation.

Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Cadmium Carbonate	Probable carcinogen	0.01 mg/ m^3 as Cd	Ingestion causes increased salivation, choking, vomiting, stomach pains and diarrhea. Inhalation may cause respiratory irritation, nausea and dyspnea.
Barbituric Acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Must be treated as potential health hazard; do not ingest.
Potassium Hydroxide	Poison Corrosive Reactive	2 mg/m^3 – Ceiling	Inhalation symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Swallowing may cause severe burns of mouth, throat and stomach. Other symptoms may include vomiting, diarrhea. Severe scarring of tissue and death may result. Contact with skin can cause irritation or severe burns and scarring. Causes irritation of eyes with tearing, redness, swelling. Greater exposures cause severe burns with possible blindness.
Chloramine T Hydrate	Poison		May be harmful by inhalation, ingestion, or skin absorption. This material is irritating to mucous membranes and upper respiratory tract. Avoid contact and inhalation.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3. Preparation of sodium hydroxide solutions produces considerable amounts of heat. Use plastic containers to mix this solution, if possible. If glass containers are used, they must be free of any cracks or irregularities.

5.4. The acidification of samples prior to extraction/preparation can result in the release of a highly toxic gas--hydrogen cyanide.

5.5. If samples are identified with cyanide concentrations equal to or greater than 200 mg/L, immediately notify the Department Manager and personnel responsible for hazardous waste shipping. Those samples must be identified as extremely hazardous for other chemists and must receive special attention during disposal.

5.6. Potassium cyanide and sodium cyanide will give off hydrogen cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea,

unconsciousness, and potentially death.

5.7. Cyanide and cyanide salts are extremely toxic. Addition of acid can generate hydrogen cyanide gas, which can be extremely dangerous.

5.8. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded. Other gloves must be cleaned.

5.9. Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.10. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents must be conducted in a fume hood with the sash closed as far as the operations will permit.

5.11. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.12. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported**immediately** to the EH&S Coordinator and to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. 2 mL and 4mL Cuvettes

6.2. 100 mL, 250 mL, 1000 mL volumetric flasks

6.3 Volumetric pipettes: various, ranging from 0.01 mL to 20 mL

6.4 Top loading balance: capable of accurately weighing± 0.01g

6.5 Balance: Analytical, capable of accurately weighing 0.0001g

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Cadmium carbonate: powder

7.1.2. Phosphate buffer: Add 136 g of potassium phosphate - monobasic (KH_2PO_4) and 2.8 g of sodium phosphate – dibasic anhydrous (Na_2HPO_4) to 800 mL of reagent water in a 1 liter volumetric flask. Mix, bring to volume with reagent water. (May be purchased commercially.)

7.1.3. Chloramine-T reagent: Add 1.0 g of chloramine-T to a 250 mL volumetric flask and dilute to volume with reagent water. Prepare fresh daily. (May be purchased commercially.)

7.1.4. Pyridine reagent: Add 15.0 g of barbituric acid to a 1 liter volumetric flask. Add 75 mL of pyridine and 15 mL of concentrated hydrochloric acid (HCl) and mix. Bring to volume with reagent water and store at 4°C ± 2°C in an amber glass bottle. The maximum shelf life is six months or the vendor's expiration date, whichever is earlier

Note: The pyridine barbituric acid may be purchased commercially. Filter 50 ml of the pyridine barbituric acid, and bring up to 250 ml with DI water.

7.1.5. Sodium Hydroxide (NaOH): reagent grade

7.1.6. 1.0 N Sodium hydroxide: Carefully add 40 g of NaOH to 800 mL reagent water. Dilute to 1 liter with reagent water.

7.1.7. 0.25 N sodium hydroxide: Add 250 mL of 1N NaOH to a 1 liter volumetric flask, and dilute to volume with reagent water.

Note: 0.25 N NaOH may be purchased instead.

7.2. Standards

7.2.1. Primary Source Cyanide Stock Standard, 1000 mg/L: Add 2.51 g of potassium cyanide (KCN) and 2.0 g of potassium hydroxide (KOH) to a 1000 mL volumetric flask and dilute to volume with reagent water. Mix well and store in glass amber container. Stable for 1-3 months. Additional information can be found in SOP NC-QA-017.

Note: This stock standard may also be purchased.

Note: Prepared stock standard must be standardized prior to use. See Appendix I (SOP NC-WC-032)

7.2.2. Secondary Source Cyanide Standard, 1000 mg/L: Follow Section 7.2.1 using an

alternate source of Potassium Cyanide (KCN).

Note: This stock standard may also be purchased.

Note: Prepared stock standard must be standardized prior to use. See Appendix I (SOP NC-WC-032).

7.2.3. Calibration Standards (Water and Solid Matrices)

7.2.3.1. Pipette the appropriate amount of cyanide standard into 100 mL volumetric and bring to volume with 0.25N NaOH. The low standard must be at, or below, the reporting limit. Prepare weekly.

Concentration CN- Calibration Standards	ML CN-	Final Volume
10 mg/L	1 mL of 1000 mg/L	100 mL Dilution from stock primary and secondary
1.0 mg/L (secondary only)	20 mL of 10 mg/L	200 mL LCS/MS
0.1 mg/L (secondary only)	1 mL of 10 mg/L	100 mL ICV
0.01 mg/L	0.1 mL of 10 mg/L	100 mL MRL check
* 0.2 mg/L	2 mL of 10 mg/L	100 mL Calibrant
0.1 mg/L	1 mL of 10 mg/L	100 mL CCV Solution

*Denotes calibration standards

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Solid samples are not chemically preserved. Water samples are preserved with NaOH to a pH >12. All samples are stored at 4°C ± 2°C in plastic or glass containers.

8.2. The holding time for samples is 14 days from sampling to analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples, excluding QC samples (Laboratory Control Sample (LCS), Method Blank (MB), Matrix Spike/Matrix Spike Duplicate (MS/ MSD)), which are processed similarly with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All

samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank

9.2.1. One Method Blank must be processed with each preparation batch. The method blank consists of reagent water or 0.25N NaOH and must contain all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The Method Blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The Method Blank must not contain any analyte of interest at, or above, the reporting limit. For Ohio VAP Projects, the Method Blank must not contain any analyte above the reporting limit.

9.2.2. A Method Blank consists of 50 mL 0.25N NaOH for Total Cyanide analysis or 50 mL reagent water for Weak Acid Dissociable and Amenable Cyanide analysis must be distilled and analyzed with each analytical batch of samples. See SOP NC-WC-032 for distillation instructions.

9.2.3. Corrective Action for Method Blanks

9.2.3.1. If the analyte level in the Method Blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and re-analyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data**must be addressed in the project narrative.**

9.2.3.2. If there is no analyte greater than the RL in the samples associated with an unacceptable Method Blank, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One Laboratory Control Sample from an independent source must be processed with each preparation batch. The Laboratory Control Sample must be carried through the entire analytical procedure. The Laboratory Control sample is used to monitor the accuracy of the analytical process. Ongoing monitoring of the Laboratory Control Sample results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. A midrange Laboratory Control Sample consisting of a 0.04 mg/L (2.0 mL of 1.0 mg/L of the secondary source to 50 mL) must be distilled and analyzed with each analytical batch of samples for Total, Amenable, and Weak Acid Dissociable Cyanide analysis. See SOP NC-WC-032 for distillation instruction.

Note: A purchased complex cyanide solution may be used instead as the midrange Laboratory Control Sample for Total Cyanide analysis only.

9.3.3. Corrective Action for Laboratory Control Samples

9.3.3.1. If any analyte is outside established control limits, the system is out of control and corrective action must occur.

9.3.3.2. Corrective action will be repreparation and re-analysis of the batch unless the client agrees that other corrective action is acceptable. For Ohio VAP samples, the Laboratory Control Samples must be redistilled, unless the Laboratory control Sample is biased high and the samples are non-detect.

9.3.3.3. The only exception is if the Laboratory Control Sample recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

9.4. Additional information on QC samples can be found in QA Policy QA-003.

9.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.5.1. One Matrix Spike/Matrix Spike Duplicate pair must be processed for each batch. A Matrix Spike (MS) is a field sample to which known concentrations of target analytes have been added. A Matrix Spike Duplicate (MSD) is a second aliquot of the same sample (spiked identically as the Matrix Spike) prepared and analyzed along with the sample and Matrix Spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of, or in addition to, Matrix Spikes/Matrix Spike Duplicates. The Matrix Spike/Matrix Spike Duplicate results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for Matrix Spike/Matrix Spike Duplicate analysis.

9.5.2. A Matrix Spike/Matrix Spike Duplicate consisting of 50 mL or 1.0 g sample and 0.04 mg/L spike (2.0 mL of 1.0 mg/L to 50 mL) must be distilled and analyzed with every batch. See SOP NC-WC-032 for distillation instructions.

9.5.3. Corrective action for Matrix Spikes/Matrix Spike Duplicates

9.5.3.1. If the analyte recovery or RPD falls outside the acceptance range, the

recovery of that analyte must be in control for the Laboratory Control Sample. If the Laboratory Control Sample recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the Laboratory Control Sample is outside limits, corrective action must be taken. Corrective action must include re-preparation and reanalysis of the batch.

9.5.3.2. If the native analyte concentration in the Matrix Spike/Matrix Spike Duplicate exceeds four times the spike level for that analyte, the recovery data is automatically flagged with a "4" in TALS.

9.6. QC Acceptance Criteria

9.6.1. Control limits are established by the laboratory as described in NC-QA-018.

NOTE: Control limits for Laboratory Control Sample and Matrix Spike/Matrix Spike Duplicate for Method 335.4 are 90% to 110%.

9.6.2. Laboratory control limits are internally generated and updated periodically unless method specified. The latest version is easily accessible via LIMs

9.7. Method Detection Limits (MDLs) and MDL Checks

9.7.1. MDLs and MDL Checks are established by the laboratory as described in SOPs CA-Q-S-006 and NC-QA-021.

9.7.2. MDLs are easily accessible via LIMs.

9.8. Nonconformance and Corrective Action

9.8.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action.

10. CALIBRATION AND STANDARDIZATION

10.1. Initial Calibration

10.1.1 The instrument is calibrated daily using the 0.2 ppm standard, which is diluted by the instrument into the following concentrations: 0.005, 0.010, 0.025, 0.050, 0.100, 0.200. The calibration is verified by using a midrange ICV. The ICV is composed of the 0.1 ppm secondary standard. The ICV must not vary from the original curve by more than $\pm$ 10%, or recalibration is required. The correlation coefficient of the original curve must be $\geq$ 0.995, or recalibration is required. The curve must not be forced through the origin. An ICB sample is analyzed after the

ICV. It cannot contain the analyte of interest above the reporting limit or recalibration is required.

10.1.1.1. Linear Regression

The linear fit uses the following functions:

$$y = ax + b$$

or

$$x = \frac{(y - b)}{a}$$

Where: y = Instrument response
x = Concentration
a = Slope
b = Intercept

10.2. Continuing Calibration

10.2.1. The run is checked every ten samples and at the end of the run using a midrange CCV to verify continued linearity. It cannot vary from the original curve by more than ± 10%, or reanalysis of all samples bracketed by the failing CCV is required. If CCVs continually fail to meet criteria, this would indicate a possible issue with the calibration standard or with the CCV standard solution, and reprpeparation and reanalyzation of the calibration curve and/or CCV solution is required. The CCV is composed of the 0.1 mg/L primary standard.

10.2.2. System cleanliness is checked every ten samples and at the end of the run using a CCB. It cannot contain the analyte of interest above the reporting limit, or reanalysis of all bracketed samples is required. If CCBs continually fail to meet criteria, immediately stop the analysis and take corrective action. Corrective action can include, but is not limited to, the following: refreshing the CCB solution, recalibration, or instrument maintenance as deemed necessary. The CCB is 0.25N NaOH.

10.3. High and Low Standard

10.3.1. The distillation technique is checked by distilling a high and low standard and comparing the values obtained to the standard curve. The method recommends that the HI/LO standards be compared to the curve with a +/-10% agreement. Re-analysis must occur if the standards do not meet criteria. The HI/LO standards are evaluated against all applicable batch QC.

10.4 Linear Calibration Range

10.4.1 The Linear Calibration Range (LCR) must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by ±10%, linearity must be re-established. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo and is approved by a Technical Specialist. The Nonconformance Memo must be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.This is not applicable for Ohio EPA/VAP.

11.3. Sample Preparation

11.3.1. See Cyanide Distillation SOP NC-WC-032.

11.3.1.1. The sample is distilled/refluxed under acidic conditions for one hour. The released HCN is trapped in 25 mL of 0.25 N NaOH solution.

11.3.2. Sample Preparation Procedure

11.3.2.1. All Solid Samples: Test each sample for the presence of sulfides using lead acetate paper. If sulfides are present, treat the sample with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution. Avoid a large excess of cadmium carbonate and long contact time in order to minimize loss by complexation or occlusion of cyanide on the precipitated material. **If any sample in a batch is treated, the Method Blank (MB) and Laboratory Control Sample (LCS) for the batch must undergo the same treatment.** Document this in LIMS.

Note: Water must be tested prior to distillation. See Cyanide Distillation SOP NC-WC-032.

11.3.3 For DoD work, refer to SOP NC-QA-016 for specific details.

11.4. Sample Analysis

11.4.1. Recommended Instrument Conditions

11.4.1.1. See manufacturer's information for operation instructions.

11.4.1.2. Perform instrument startup.

11.4.1.3. Add reagents to the Konelab reagent wheel.

11.4.1.4. Insert standards and samples to autosampler segments.

11.4.2. Sample Analysis Procedure

11.4.2.1. See manufacturer's information for operating instructions.

11.4.2.2. Calibrate the instrument (see section 10.1.1). The correlation coefficient must be > 0.995 to continue.

11.4.2.3. The ICV (from the secondary source) and the ICB are analyzed after the calibration and followed by an MRL check. CCVs (from the primary source) and CCBs are analyzed between every ten samples and at the end of the run.

11.4.2.4. Sample distillates higher than the highest calibration standard (0.2 mg/L) must be diluted with 0.25 N NaOH and re-analyzed.

11.4.2.5. Any samples analyzed after a high sample must be re-analyzed if carryover is suspected.

11.5. Analytical Documentation

11.5.1. Record all analytical information appropriately in LIMS, including the analytical data from standards, Method Blanks, Laboratory Control Samples, Matrix Spike/Matrix Spike Duplicates, and any corrective actions or modifications to the method.

11.5.2. All standards and reagents are logged in the LIMS standards and reagents module. All standards are assigned a unique number for identification.

11.5.3. Documentation such as all associated instrument printouts (final runs, screens,

reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4. Sample results and associated QC are transferred directly into LIMS from the instrument. Level I and Level II reviews are done in LIMS.

12. DATA ANALYSIS AND CALCULATIONS

12.1. $$\textit{Total Cyanide, mg / L} = \frac{\textit{mg / L } CN^-}{\textit{mL of sample distilled}} \frac{\textit{x 50}}{} \textit{x D}$$

12.2. $$\textit{Total Cyanide, mg / kg} = \frac{\textit{mg / L } CN^- \quad \textit{x 50}}{\textit{g of sample distilled.}} \textit{x D}$$

12.3. $$\textit{Amenable Cyanide, mg / L} = \textit{Total } CN^- \textit{ (mg / L) - Chlorinated } CN^- \textit{ (mg / L)}$$

Where:
mg/L = can also be mg/kg

D = Dilution Factor = $$\frac{\textit{Final Volume of Dilution}}{\textit{Volume of Sample Distillate Used}}$$

Note: Weak Acid Dissociable Cyanide has the same calculations as Total Cyanide

12.4. Laboratory Control Sample (LCS) Recovery:

$$\frac{\textit{Instrument Value}}{\textit{0.04(true)}} \textit{ x 100 = \% Recovery}$$

Note: The true value may vary by manufacturer or analysis.

12.5. CCV Recovery:

$$\frac{\textit{Instrument Value}}{\textit{0.1 (true)}} \textit{ x 100 = \% Recovery}$$

NOTE: *CCV recovery must be between 90-110% for data to be acceptable. If CCV recovery is not within these limits, re-analysis is required.*

12.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Recovery for Waters and solids

$$\frac{A - B}{0.040\ (true)} \times 100 = \%\ Recovery$$

Where:

A = Instrument value Matrix Spike/Matrix Spike Duplicate (MS/MSD)

B = Sample instrument value

12.7 Additional equations and calculations are liste d in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".Waste Management

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local laws and

regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2. Waste Streams Produced by the Method

15.2.1. The waste from Konelab analysis is collected in the waste bucket housed in the instrument. The waste container is emptied at the end of the day in the "Acid Waste" container.

15.2.2. Filter paper contaminated with cadmium sulfide complex. This waste is placed in a container labeled "Solid Waste".

15.2.3. Aqueous rinsates from distillation tube clean up. This waste is collected in the lab and disposed of in a container labeled "Acid Waste".

15.2.4. Standard Waste and High Concentration Samples: This waste is disposed of in the designated container labeled "High Cyanide/Basic Waste." NO ACID is added to this container.

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks followed by annual refresher training.

16. REFERENCES

16.1. References

16.1.1. SW846, Test Methods for Evaluating Solid Waste Method 9012A, Revision 1, 1996.

16.1.2. SW846, Test Methods for Evaluating Solid Waste, Method 9012B, Revision 2, 2004.

16.1.3. EPA 600; Cyanide, Total and Cyanide, Amenable to Chlorination; Methods 335.2, March 1983

16.1.4. EPA 600; Determination of Total Cyanide by Semi-Automated Colorimetry, 335.4, Revision 1.0, August 1993

16.1.5. EPA 600 Cyanides, Amenable To Chlorination (Titrimetric, Spectrophotometric), Method 335.1, 1974

16.1.6. Standard Methods for the Examination of Water and Wastewater, 1999: Total, Amenable, and Weak Acid Dissociable Cyanide; Methods 4500-CN-E, 4500-CN-I, and 4500-CN-G

16.1.7. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.8. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan current version

16.1.9. Corporate Quality Management Plan (CQMP), current version

16.1.10. Ohio EPA Laboratory Manual for Chemical Analyses of Public Drinking Water, 2000

16.1.11. Revision History

Historical File:		Revision 5: 06/24/9		Revision 8.1: 05/30/0
		Revision 6: 06/28/9		Revision 8.2: 12/30/0
		Revision 7: 05/31/01		Revision 8.3: 06/15/1
		Revision 8: 11/08/0		Revision 8.4-A: 04/16/12

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-016

16.2.6. Cyanide Preparation Method, NC-WC-032

16.2.7. Standards and Reagents, NC-QA-017

16.2.8. Selection of Calibration Points, CA-T-P-002

16.2.9. Calibration Curves (General), CA-Q-S-005

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. The reporting limit (RL) is 0.01 mg/L for waters (50 mL used) and 0.50 mg/kg for solids (1.0 g used). The lowest level of the calibration curve can be used as the reporting limit upon request.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Method Deviation

17.2.1. Method of Standard Addition is not performed for samples with matrix interference (sulfides).

17.2.2. Method 9012A states that the CCV must be within ±15%, or recalibration is required. The laboratory reanalyzes all samples bracketed by CCVs that are outside of ±10%, and recalibrates only if deemed necessary by continual failures.

17.2.3. EPA Method 335.4 calls for the samples to distill/reflux for 1.5 hours. The laboratory distills/refluxes for 1 hour.

Appendix I

Glossary of Acronyms

CCB Continuing Calibration Blank
CCV Continuing Calibration Verification
CQMP Corporate Quality Management Plan
DOC Demonstration of Capability
DOD Department Of Defense
DUP Duplicate
EH&S Environmental Health and Safety
ICB Initial Calibration Blank

ICV Initial Calibration Verification
LCR Linear Calibration Range
LCS Laboratory Control Sample
LIMS Laboratory Information Management System
MB Method Blank
MDL Method Detection Limit
MS Matrix Spike
MSD Matrix Spike Duplicate
MSDS Material Safety Data Sheet
NCM Non Conformance Memo
OSHA Occupational Safety and Health Administration
OVAP Ohio Voluntary Action Program
PEL Permissible Exposure Limit
QAM Quality Assurance Manual
QA/QC Quality Assurance/Quality Control
RPD Relative Percent Difference
SOP Standard Operational Procedure
STEL Short Term Exposure Limit
WAD Weak Acid Dissociable

TestAmerica Canton

SOP Amendment Form

SOP NUMBER: **NC-WC-004, Rev. 3.5 Effective date 4/23/12**

SOP TITLE: Total Solids, Percent Moisture, and Total Settleable Solids

REASON FOR ADDITION OR CHANGE: Addition noted below

CHANGE EFFECTIVE FROM: (DATE): 11/21/13

Change(s) Made: Added the following to the SOP:

11.5.4 Allow the sample to settle for forty five minutes, gently agitate sample near the sides of the cone with a glass stir-rod. Then allow the sample to settle for another fifteen minutes.

EDITED BY/DATE: Lucas Grossman 11/21/13

*APPROVED BY:

Lucas Grossman

Technical Reviewer Signature Date: 11/21/13

Rebecca Strait

QA Manager Signature Date: 11/21/13

Technical Director Signature Date: 11/22/13

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Canton

SOP No. NC-WC-004, Rev. 3.5
Effective Date: 04/23/12

Title: TOTAL SOLIDS, PERCENT MOISTURE, AND TOTAL SETTLEABLE SOLIDS,

[Method: EPA Methods 160.3 Modified, EPA 160.5, and ASTM D2216-98 and]

Approvals (Signature/Date):

[redacted] Technology Specialist	04/18/12 Date	[redacted] Health & Safety Coordinator	04/19/12 Date
[redacted] Quality Assurance Manager	04/23/12 Date	[redacted] Laboratory Director	04/18/12 Date

This SOP was previously identified as SOP No. NC-WC-0004, Rev 3.4, dated 12/22/09

Facility Distribution No. _____ **Distributed To: _____**

TABLE OF CONTENTS

1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Total Solids; Percent Moisture; and Settleable Solids; in wastewaters, sludges, and solids. It is based on EPA 160.3 Modified, EPA 160.5, and ASTM D2216-98,. The approximate working range for Total Solids is 10 to 100% for non-waters.

1.2. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. A homogenous sample is dried at 104 °C ± 1°C, and the difference in the weight loss of the sample represents the Total Solids.

2.2. Settleable Solids. Settleable matter is measured volumetrically with an Imhoff cone.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Samples high in concentrations of minerals must be dried longer, desiccated, and weighed quickly to prevent any excess weight added due to absorption of water from the atmosphere.

4.3. Non-homogeneous samples may give erratic results.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Oily samples or those that contain volatile chemicals may ignite during this procedure. In the case of a fire, the oven should be turned off and allowed to cool before the sample can be removed and put under a hood.

5.3. There are no materials used in this method that have a significant or serious hazard rating. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable**. Therefore, unless they are known to be non-hazardous, all samples must be opened, transferred, and prepared in a fume hood or under other means of mechanical ventilation. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and to a Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Drying Oven

6.2. Desiccators

6.3. Evaporating dishes: various

6.4. Tongue blades

6.5. Sample containers

6.6. Top loading balance: capable of accurately weighing ± 0.01 g

6.7. Analytical balance: capable of accurately weighing ±0.0001 g

6.8. Beakers: glass, various

6.9. Volumetric flasks: various

6.10. Imhoff cones

6.11. Labels

6.12. Graduated Cylinder, 1000 mL, 10mL, Class A

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Reagent water

7.1.2. Sand

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored in plastic or glass containers at 4°C ± 2°C.

8.3. The holding time is 48 hours for settleable solids.

8.4. There is no recommended holding time for non-water samples.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, sample duplicate) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank

9.2.1. One method blank (MB) must be processed with each preparation batch of Settleable Solids samples. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

NOTE: There is no method blank applicable for Total Solid or Percent Moisture.

9.2.2. Corrective Action for Blanks

9.2.2.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are re-prepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.2.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.3. Laboratory Control Sample

9.3.1. For the Settleable Solids method, an LCS sample is required.

9.3.1.1 One mL of sand will be added to a 10 mL graduated cylinder. 1000 mL of water is added to a liter container. The sand mixture is added to the liter container and mixed. The resulting mixture is transferred to an Imhoff cone and allowed to settle for one hour. Control limits will be 90-110%.

9.4. Duplicates

9.4.1. A sample duplicate (DU) is a second aliquot of an environmental sample, taken from the same sample container when possible, that is processed with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. The sample and DU results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample/DU precision results are not necessarily representative of the precision for other samples in the batch.

9.4.2. Sample duplicates are performed at a frequency of 10% and must meet laboratory-specific limits for precision.

9.4.3. Sample duplicates are not applicable for Settleable Solids.

9.5. Control Limits

9.5.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.5.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMs.

9.6. Method Detection Limits (MDLs) and MDL Checks

9.6.1. MDLs and MDL Checks are established by the laboratory as described in SOPs CA-Q-S-006 and NC-QA-021.

9.6.2. MDLs are easily accessible via LIMs.

9.7. Nonconformance and Corrective Action

9.7.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action.

10. CALIBRATIONAND STANDARDIZATION

10.1. Not applicable

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.3. Total Solids (Percent Moisture)

11.3.1. For solid and sludge samples, label and weigh an evaporating dish on an analytical balance. Record the weight in LIMS. For solid samples (TS), a universal tare weight is recorded in LIMS. For solid samples, Sample Receiving will divide the soil sample into a pre-tared container with a label. Weigh and record the wet weight in LIMS.

11.3.2. Sample Duplicate for solid samples (TS), Sample Receiving will supply the sample duplicate. If the sample duplicate is not supplied from Receiving, split some sample off from another container into a new container with a label. Weigh and record the weight in LIMS.

11.3.3. Place dishes with sample in the drying oven (103° - 105°C) until dry (minimum of 12 hours). Document the time samples were placed in the oven in LIMS.

11.3.4. Remove the samples from the drying oven when they are dry. Document the time samples were removed from the oven in LIMS.

11.3.5. Place the dishes in the desiccator for at least one hour. Weigh the sample and dish on the top loading balance. Record the weight in LIMS.

11.4. Percent Moisture

11.4.1. Follow Section 11.3.

11.5. Settleable Solids

11.5.1. A method blank is prepared from one liter of reagent water. A blank must be analyzed with each batch of 20 or less samples to ensure Imhoff cones are clean and free of particulate contamination. An LCS sample must be analyzed as noted in Section 9.3.

11.5.2. Shake one liter of sample vigorously for ten seconds.

11.5.3. Pour the sample into an Imhoff cone, filling the cone to the one-liter mark.

11.5.4. Allow the sample to settle for forty five minutes, gently agitate sample near the sides of the cone with a glass stir-rod. Then allow the sample to settle for another fifteen minutes.

11.5.5. Measure the volume of settleable solids by observing the location of the border between the settled matter and the supernatant liquid.

11.5.5.1. If pockets of liquid are trapped beneath large settled particles, estimate the volume of the pockets and subtract from the total measured volume.

11.6. Analytical Documentation

11.6.2. All standards are logged into the LIMS standards and reagents module. All standards are assigned a unique number for identification.

11.6.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.6.4. Sample results and associated QC are entered into LIMs. Level I and Level II technical reviews are done in LIMS..

12. DATA ANALYSIS AND CALCULATIONS

12.1. Total Solids

$$\textit{Total Solids, \% (non-waters)} = \frac{(A - B)\ x\ 100}{(C - B)}$$

Where: A = Final weight of dried sample and dish, g
B = Initial weight of dish, g
C = Initial weight of wet sample and dish, g

12.2. Dry Weight

$$\textit{Dry Weight} = \frac{\textit{Sample Test Result x 100}}{\textit{(\%) Total Solid Results}} = \textit{Dry Weight}$$

12.3. Percent Moisture

$$\textit{\%Moisture} = 100 - \textit{\%TS (Section 12.1)}$$

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications

13.2.1. The Group/Team Leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method

15.2.1. The following waste streams are produced when this method is carried out.

15.2.1.1. Acidic sample waste generated by the analysis. This waste is collected in the laboratory in a designated container identified as "Acid Waste".

15.2.1.2. Contaminated filter and filter residue generated by the analysis. This waste is collected in the laboratory in a designated container identified as "Solid Waste".

15.2.1.3. Weighing containers and filter residue generated by solid sample analysis. This waste is collected in the laboratory in a designated container identified as "Solid Waste".

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks followed by an annual refresher training.

16. REFERENCES

16.1. References

16.1.1. EPA 600, 1984, Total Residual Solids, Method 160.3

16.1.2. EPA Settleable Solids, Method 160.5

16.1.3. Annual Book of ASTM Standards, Volume 04.08, 1990

16.1.4. Corporate Quality Management Plan (CQMP), current version

16.1.5. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.6. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.7. Ohio Bureau of Underground Storage Tank Regulations (BUSTR) Technical Guidance Manual, April 2005

16.1.8. Revision History

Historical File:		Revision 3.0: 08/04/00		Revision 3.3: 09/10/07
		Revision 3.1: 11/06/04		Revision 3.4: 12/22/09
		Revision 3.2: 02/02/06		

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection Limits, CA-Q-S-006 and NC-QA-021

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-016

16.2.6. Standards and Reagents, NC-QA-017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. The reporting limit for Method 160.3 is 10%. The reporting limit for Method 160.5 is 0.1 ml/L/hr. The reporting limit for Percent Moisture by ASTM D2216-98 is 0.1%.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

TestAmerica
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TestAmerica Canton

SOP No. NC-WC-093, Rev. 1
Effective Date: 9/19/13

Title: TOTAL, CARBONATE, BICARBONATE, AND HYDROXIDE ALKALINITY

[Method: SM2320B and EPA 310.1]

Approvals (Signature/Date):

Technology Specialist	09/16/13 Date	Health & Safety Coordinator	09/19/13 Date
Quality Assurance Manager	09/16/13 Date	Laboratory Director	09/16/13 Date

This SOP was previously identified as SOP NC-WC-093, Rev 0 dated 05/27/11

Copyright Information:

Facility Distribution No. _____ **Distributed To: _____**

TABLE OF CONTENTS

1. SCOPE AND APPLICATION

1.1. This method is applicable for the determination of total, carbonate, bicarbonate, and hydroxide alkalinity in surface, saline, domestic, and industrial waters and wastewaters. It is also applicable to the determination of water-soluble alkalinity in solid samples if they have been prepared according to NC-IP-009. It is based on EPA Method 310.1 and Standard Method 2320B. The working linear range is 5 to 2500 mg/L.

1.2. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. An unaltered sample is titrated to an electrometrical endpoint of pH 4.5. **The sample must not be filtered, concentrated, or altered in any way.** An unaltered sample is tested for alkalinity, and calculations are performed to determine carbonate (CO_3-2), bicarbonate (HCO_3-), and/or hydroxide (OH^-).

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica Canton Laboratory Quality Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Samples with salts of weak organic and inorganic acids and greases or oils will interfere with pH measurements.

4.3. The method is suitable for all concentration ranges of alkalinity; however, appropriate aliquots should be used to avoid a titration volume greater than 50 mL.

4.4. Suspended solids or precipitates may coat the electrode and cause a sluggish response.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3. Eye protection, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Alkalinity – Manual (Appendix A)

6.1.1. Stir plate and stir bars

6.1.2. Graduated cylinders: various

6.1.3. Beakers: various

6.1.4. Buret: Class A 25 mL or 50 mL (preferred)

6.2. Alkalinity - Automated

6.2.1. Autotitrator

6.2.2. 50 mL centrifuge tubes

6.3. Alkalinity – Manual and Automated

6.3.1. pH meter and electrode(s) with temperature compensation

6.3.2. Volumetric pipettes: various

6.3.3. Autopipettor and disposable tips

6.3.4. Top loading balance: Capable of accurately weighing ±0.01 g

6.3.5. Volumetric flasks: various

6.3.6. Oven

6.3.7. Desiccator

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. 0.02 N Sulfuric Acid: reagent grade, purchased, standardized monthly.

7.1.2. Sodium Carbonate (Na_2CO_3): standard grade, dry overnight in 180°C oven and cool in a desiccator, or purchased primary standard grade.

7.1.3. Sodium Carbonate Solution: Add 0.20g of Na_2CO_3 (record exact weight of Na_2CO_3 used) to a 250mL volumetric flask and dilute to volume with reagent water. Mix well.

7.2. Standards

7.2.1. Target Calibration Standard

7.2.1.1. pH Buffers: 4, 7, and 10 (manufactured)

7.2.2. Laboratory Control Sample

7.2.2.1. Alkalinity Standard, 25,000 mg/L $CaCO_3$, purchased or other commercially available reference solutions

7.2.3. Matrix Spike Standard

7.2.3.1. Alkalinity Standard, 25,000 mg/L $CaCO_3$, purchased

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored in plastic or glass containers at 4°C ± 2°C.

8.3. The holding time is 14 days from sampling to analysis.

8.4. The bottle must be filled with no headspace and provided in a separate container.

8.5. Do not open sample bottle before analysis. If other tests are to be performed from the same bottle, Alkalinity must be determined first. This is dependent on the client sending a separate bottle for alkalinity.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD, or Sample Duplicate) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank (MB)

9.2.1. One MB must be processed with each preparation batch. The MB consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The MB should not contain any analyte of interest at or above the reporting limit.

9.2.2. An MB consisting of 50 mL of reagent water and all other reagents added to samples within the analytical batch is analyzed with each analytical batch of samples.

9.2.3. Corrective Action for MBs

9.2.3.1. If the analyte level in the MB exceeds the reporting limit for the analytes of interest in the sample, all associated samples are re-prepared and re-analyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.3.2. If there is no analyte greater than the RL in the samples associated with an unacceptable MB, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS) for Total Alkalinity Only

9.3.1. One aqueous LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. An LCS consisting of 1mL of the 25,000 mg/L alkalinity standard and 50 mL reagent water or other commercially available reference solution is analyzed with each analytical batch of samples.

9.3.3. Corrective Action for LCS

9.3.3.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.3.2. The only exception is that if the LCS recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

9.3.3.3. Corrective action will be re-preparation and re-analysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) for Total Alkalinity Only

9.4.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. An MS/MSD consisting of 1 mL of the 25,000 mg/L alkalinity standard and 50 mL of the sample will be analyzed.

9.4.3. Corrective action for MS/MSDs

9.4.3.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.4.3.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported and flagged with a "4" in LIMS

9.4.3.3. If client program requirements specify to confirm matrix interferences, re-preparation and re-analysis of the MS/MSD may be necessary.

9.5. Sample Duplicates (DU) for Total Alkalinity Method 2320B, Carbonate, Bicarbonate, and Hydroxide Alkalinity

9.5.1. DUs are performed at a minimum frequency of one per 10 analytical samples and must meet laboratory-specific limits for precision.

9.6. QC Acceptance Criteria

9.6.1. Control limits are established by the laboratory as described in NC-QA-018.

9.6.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are listed in the TestAmerica Canton Quality Assurance Manual (QAM) and the latest version is easily accessible via LIMs.

9.7. Method Detection Limits (MDLs) and MDL Checks

9.7.1. MDLs and MDL Checks are established by the laboratory as described in SOPs CA-Q-S-006 and NC-QA-021.

9.7.2. MDLs are listed in the TestAmerica Canton Quality Assurance Manual (QAM) and the latest version is easily accessible via LIMs.

9.8. Nonconformance and Corrective Action

9.8.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action.

10. CALIBRATION AND STANDARDIZATION

10.1. Instrument Directions

10.1.1. Calibrate the pH meter according to the manufacturer's specifications. See pH Electrode Method SOP NC-WC-010 if measuring for alkalinity manually.

10.2. Initial Calibration

10.2.1. The pH meter is calibrated everyday with the 4, 7, and 10 calibration buffers and is verified at the beginning of the run by using the 7 buffer. The pH buffers should bracket the sample concentration.

10.3. Continuing Calibration

10.3.1. The pH meter is checked every ten readings with a midrange (pH 7) buffer to ensure the calibration remains linear. The acceptance range for the calibration check is 7 ± 0.05 pH units or recalibration is necessary.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a root cause and corrective action described.

11.3. Sample Preparation

11.3.1. For solids preparation, see SOP NC-IP-009.

11.3.2. No preparation is necessary for water samples.

11.4. Standardization

11.4.1. To standardize 0.02 N sulfuric acid, titrate 50 mL of the sodium carbonate solution with 0.02 N H_2SO_4 to a pH of 4.5. This should be performed monthly or on a new lot of acid (whichever is more frequent). Calculate as follows:

$$N = \frac{A \times 1000}{53.00 \times B} \quad \text{(Manual)}$$

$$N = \frac{A \times 1000}{53.00 \times B} \times \frac{30}{50} \text{ (Autotitrator)}$$

Where: A = gNa_2CO_3

B = mL 0.02 N H_2SO_4 titrant

11.5. Repeat standardization two or three more times. Record the standardization as an intermediate reagent for the Sulfuric acid in the LIMS Reagents module.

11.6. Sample Analysis – Manual

11.6.1. See Appendix A for the manual sample analysis procedure.

11.7. Sample Analysis – Automated – Summary

11.7.1. The samples are analyzed on the autotitrator for Alkalinity.

11.7.2. Do not shake sample.

11.7.3. Samples should be analyzed at room temperature

11.7.4. Place 50 mL of sample or an aliquot diluted to 50 mL with reagent water, in a 50 mL centrifuge tube. See Manufacturer's information for operating instructions.

11.7.5. If a dilution of the sample was done, change the volume on the schedule to reflect the dilution (based on a 30 mL sample inject). For alkalinity, the dilution factor will be taken into account in the final calculation. Do not manually multiply the dilution unless it was not typed into the schedule.

11.7.6. After the results have been gathered from the instrument make sure to check the pH of all the samples. If a sample has an initial pH of >4.5 and the Total Alkalinity is zero, the sample must be diluted and re-analyzed.

11.8. Analytical Documentation

11.8.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.8.2. Record all standards and reagents in the LIMS Reagents module. All standards and reagents are assigned a unique number for identification.

11.8.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.8.4. Record all sample results and associated QC into LIMS. Level and Level II reviews are performed in LIMS.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Calculations

12.1.1. Alkalinity, mg/L $CaCO_3$ to pH 4.5 = $\frac{A \times N \times 50{,}000}{mL\ of\ sample}$

12.1.2. LCS % = $\frac{mg/L}{500\ (true)} \times 100$

MS/MSD % = $\frac{B - C}{500\ (true)} \times 100$

Where:

A = mL of titrant
N = Normality of titrant
B = MS/MSD, mg/L
C = Sample, mg/L

12.1.3 Calculate the alkalinity according to the total alkalinity method.

12.1.4 Calculate the following alkalinities using the total alkalinity value.

ALKALINITY RELATIONSHIPS

Result of Titration	Hydroxide Alkalinity as $CaCO_3$	Carbonate Alkalinity as $CaCO_3$	Bicarbonate Concentration as $CaCO_3$
P = 0	0	0	T
P < ½ T	0	2P	T – 2P
P = ½ T	0	2P	0
P > ½ T	2P – T	2 (T – P)	0
P = T	T	0	0

P = Phenolphthaleinalkalinity
T = Total alkalinity

12.5 Phenolphthaleinalkalinity is calculated when titrating to an endpoint of 8.3, and is performed by the instrument.

12.6 Calculation of alkalinity relationships: see the referenced methods for these calculations.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications

13.2.1. The Group/Team Leader has the responsibility to ensure an associate who has been properly trained in its use and has the required experience performs this procedure.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method

15.2.1. The following waste streams are produced when this method is carried out.

15.2.1.1. This waste is drained from the titration cell into the aqueous waste stream since the pH range is between 5 and 10. Any sample waste generated that is not in this pH range is collected in a designated container identified as "Acid Waste".

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica

Canton. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks followed by annual refresher training.

16. REFERENCES

16.1. References

16.1.1. EPA-600/4-79-020, Methods for Chemical Analysis of Water and Wastes, Revised March 1983, Alkalinity, Method 310.1

16.1.2. Standard Methods for the Examination of Water and Wastewater, Alkalinity Methods, 2320B, 1997.

16.1.3. EPA 600, Methods for Chemical Analysis of Water and Wastes, pH, Method 150.1

16.1.4. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.5. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.6. Corporate Quality Management Plan (CQMP), current version

16.1.7. Revision History

Historical File: (formerly NC-WC-003 and NC-WC-006)		(-003) Rev 1: 05/16/95 (-006) Rev 2: 03/04/98		Rev 0: 05/27/11
		(-003) Rev 2: 03/20/00 (-006) Rev 3: 04/04/00		
		(-003) Rev 2.1: 02/08/01 (-006) Rev 4: 02/06/01		
		(-003) Rev 2.2: 12/07/04 (-006) Rev 5: 10/25/04		
		(-003) Rev 2.3: 11/09/07 (-006) Rev 6: 01/30/06		
		(-006) Rev 7: 03/01/08		

16.2. Associated SOPs

16.2.1. Solid Extraction for Wet Chemistry Parameters, NC-IP-009

16.2.2. pH Electrode Method for Wet Chemistry Parameters, NC-WC-010

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. QA Policy, QA-003

16.2.5. Glassware Washing, NC-QA-014

16.2.6. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.7. Navy/Army SOP, NC-QA-016

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. The lower reporting limit (RL) for undiluted samples is 5 mg/L $CaCO_3$.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Method Deviation

17.2.1. A fixed endpoint of 4.5 is used for all samples since the sample concentration is often unknown.

17.2.2. The Sodium Carbonate (Na_2CO_3) is dried at 180°C overnight instead of at 250° C for 4 hours.

17.2.3. The standard acid solution is not boiled for 3-5 minutes under a watch glass cover.

17.2.4. A sample dilution will be needed if the 4.5 endpoint cannot be reached after adding the maximum amount of acid.

17.2.5. Low alkalinity samples will also be determined by the method above, and not by the low-alkalinity method.

17.2.6. All Alkalinity samples will be titrated with 0.02N sulfuric acid.

17.3. Appendix A: Manual procedure for determining Alkalinity

17.3.1. The manual procedure is to be used only when absolutely required in order to meet sample hold times.

17.3.2. Sample Analysis – Manual

17.3.2.1. Do not shake sample.

17.3.2.2. Record the initial pH prior to sample analysis.

17.3.2.3. Use a sufficiently large volume of titrant (>20 mL in a 50 mL buret) to obtain good precision while keeping the volume low enough to permit a sharp end point

17.3.2.4. Place 50 mL of sample or an aliquot diluted to 50 mL with reagent water, in a beaker. Begin mixing; measure and record initial pH of the sample. Titrate the sample to an endpoint of pH 4.5 with 0.02 N H_2SO_4. Record the volume of the titrant in LIMS. Samples requiring >50 mL titrant (> 75 mL using autotitrator) should be re-analyzed using less sample volume. Record any dilutions in LIMS.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Canton

SOP No. NC-WC-084, Rev. 12
Effective Date: 08/28/13

Title: DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY

[Method: EPA Method 300.0A and SW846 Method 9056A]

Approvals (Signature/Date):

Technology Specialist	08/22/13 Date	Health & Safety Coordinator	08/21/13 Date
Quality Assurance Manager	08/21/13 Date	Laboratory Director	08/24/13 Date

This SOP was previously identified as SOP No. NC-WC-084, Rev 11, dated 1/10/12

Facility Distribution No. _____ **Distributed To: _____**

TABLE OF CONTENTS

1. SCOPE AND APPLICATION

1.1. This method covers the determination of fluoride, chloride, nitrite, bromide, nitrate, ortho-phosphate and sulfate in surface waters, mixed domestic, and industrial wastewaters, groundwater, reagent waters, solids (after extraction Section 11.10) and leachates (when no acetic acid is used). This SOP is based on EPA Method 300.0A and SW846 9056A.

1.2. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. A 25 µL volume of sample is introduced into the ion chromatograph. The sample is pumped through two different ion exchange columns, then a suppressor device and into a conductivity detector. The first two columns, a pre-column or guard column and a separator column, are packed with low-capacity, strongly basic anion exchange resin. Ions are separated into discrete bands based on their affinity for the exchange sites of the resin. The last column is a suppresser column that reduces the background conductivity of the eluent to a low or negligible level and converts the anions in the sample to their corresponding acids. The separated anions in their acid form are measured using an electrical conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current version

4. INTERFERENCES

4.1. Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.

4.2. The water dip or negative peak that elutes near, and can interfere with, the fluoride peak can usually be eliminated by the addition of .05 mL of concentrated eluent to each 5ml vial of standard and sample.

4.3. Method interferences may be caused by contaminants in the reagent water, reagents, glassware and other sample processing apparatus that lead to discrete artifacts or an elevated baseline in the ion chromatograms.

4.4. Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known co-elution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant; however, it is the responsibility of the user to generate precision and accuracy information in each sample matrix.

4.5. The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

5. SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2 Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.3 A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

5.4 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.5 Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore; unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation when possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.6 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.7 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1 Balance: Analytical, capable of accurately weighing to the nearest 0.0001 g and top-loading capable of accurately weighing to the nearest 0.01 g

6.2 Ion Chromatograph: Analytical system complete with ion chromatograph and all required accessories including analytical columns, compressed gases and detectors.

6.2.1 Anion guard column: A protector of the separator column. If omitted from the system the retention times will be shorter. Usually packed with same substrate as the separator column. 4 x 50 mm, Dionex IonPac AG14 P/N 46134, or 4 x 50mm, Dionex IonPac AG19 P/N 062887, or equivalent.

6.2.2 Anion separator column: The separation shown in Figure 1 was generated using a 4 x 250 mm Dionex IonPac AS14 column (P/N 46124). The separation shown in Figure 2 was generated using a 4 x 250mm Dionex IonPac AS19 column (P/N 062885) Equivalent column may be used if comparable resolution is obtained, and the requirements of Section 9.2 can be met.

6.2.3 Anion suppressor device: Dionex anion micro membrane suppressor ERS 500 Suppressor (4mm) P/N 082540 or equivalent.

6.2.4 Detector -- Conductivity cell: Approximately 1.25 uL internal volume, Dionex, or equivalent.

6.2.5 Dionex-ChromeleonData Chromatography software or equivalent.

6.2.6 Chrom software for data processing.

6.3 Pipettes: Ranging from 0.01mL to 20mL

6.4 Volumetric Flasks: 10mL, 200mL

6.5 Filters: 0.45um

6.6 20 L container

6.7 Syringes: 10mL

6.8 Polyvials with caps

7. REAGENTS AND STANDARDS

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is

of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Reagent water. Distilled or deionized water, free of the anions of interest. Water should contain particles no larger than 0.20 microns.

7.3 Cleaned Ottawa Sand

7.3.1 Weigh 100 g of Ottawa sand and place in a 500 mL Erlenmeyer flask. Bring up to volume with DI water. Using a clean stir bar, stir for one hour. Allow to settle. Decant.

7.3.2 Bring up to volume with DI again, stir for one hour, settle, decant

7.3.3 Repeat Step 7.3.2.

7.3.4 Place sand in a clean beaker and let sit overnight in a 104? C oven.

7.3.5 Store covered in a desiccator until needed.

7.4 Helium gas and regulator

7.5 Eluent solution for DX120 and DX-320: Sodium bicarbonate (CASRN 144-55-8) 1.0 mM, sodium carbonate (CASRN 497-19-8) 3.5 mM. Dissolve 1.680 g sodium bicarbonate ($NaHCO_3$) and 7.417 g of sodium carbonate (Na_2CO_3) in reagent water (7.2) and dilute to 100 mL in a volumetric flask. Take 10 mL of this concentrated eluent solution and dilute to 2 L for use as the working eluent solution or dissolve the entire bicarbonate/carbonate amount in 20 L of reagent water. Degas for a minimum of half an hour (but not to exceed one hour) with Helium gas. Eluent for the ICS2100 is generated using a Dionex EGC III KOH eluent generator cartridge (P/N 074532), and high purity deionized water. This instrument allows the use of a gradient so the concentration of the eluent can change throughout the run.

7.6 Stock solutions (1,000 mg/L): All stocks are purchased from commercial sources. Primary and secondary sources are required for each target analyte.

7.6.1 Commercial stock solution A: F^- - 25 mg/L, Cl^- - 500 mg/L, Br^- - 100 mg/L, NO_3^- - N- 25 mg/L, PO_4– P - 25 mg/L, SO_4^{-2} - 500 mg/L. The stock solution may also be at the following concentrations: F^- - 125 mg/L, Cl^- - 2500 mg/L, Br^- - 500 mg/L, NO_3^- - N- 125 mg/L, PO_4– P - 125 mg/L, SO_4^{-2} - 2500 mg/L.

7.6.2 Commercial stock solution B: NO_2^- - N- 25 mg/L. The spike solution may also be at 125 mg/L.

7.6.3 Commercial IC Spike solution A: F^- - 125 mg/L, Cl^- - 2500 mg/L, Br^- - 500 mg/L, NO_3^- - N- 125 mg/L, PO_4– P - 125 mg/L, SO_4^{-2} - 2500 mg/L

7.6.4 Commercial IC Spike solution B: NO_2^- - N- 125 mg/L

7.7 Working standards: Prepare Cal standard #9 by mixing 1.6 mL commercial stock A, 1.6 mL commercial stock B, and 16.8 mL of eluent; or if the less concentrated solution is used, by mixing 8.0 mL A, 8.0 mL B, and 4.0 mL of eluent.

7.7.1 In 5 mL polyvials prepare the following calibration standards in eluent. Final concentrations of working standards are shown below.

7.7.1.1 Calibration Standard #1: Take 25.0 ?L of calibration standard #9, and add 4.975 mL of eluent.

7.7.1.2 Calibration Standard #2: Take 125 ?L of calibration standard #9, and add 4.875 mL of eluent.

7.7.1.3 Calibration Standard #3: Take 250 ?L of calibration standard #9, and add 4.75 mL of eluent.

7.7.1.4 Calibration Standard #4: Take 0.5 mL of calibration standard #9, and add 4.5 mL of eluent.

7.7.1.5 Calibration Standard #5: Take 1.25 mL of calibration standard #9, and add 3.75 mL of eluent.

7.7.1.6 Calibration Standard #6: Take 2.00 mL of calibration standard #9, and add 3.00 mL of eluent.

7.7.1.7 Calibration Standard #7: Take 2.50 mL of calibration standard #9, and add 2.50 mL of eluent.

7.7.1.8 Calibration Standard #8: Take 3.75 mL of calibration standard #9, and add 1.25 mL of eluent.

Calibration Standards

Level	Fluoride	Nitrite	Nitrate	Chloride	Sulfate	Bromide	Ortho-Phosphate
1	0.05	0.05	0.05	1	1	0.2	0.05
2	0.25	0.25	0.25	5	5	1	0.25
3	0.5	0.5	0.5	10	10	2	0.5
4	1	1	1	20	20	4	1
5	2.5	2.5	2.5	50	50	10	2.5
6	4	4	4	80	80	16	4
7	5	5	5	100	100	20	5
8	7.5	7.5	7.5	150	150	30	7.5
9	10	10	10	200	200	40	10

7.7.2 Prepare or purchase a secondary stock standard(s) using a standards source other than that used for the primary standards as described in Section 7.5. Dilute these stock standards as indicated in the table below to prepare the mixture to be used for the laboratory control sample (LCS) and continuing calibration verification (CCV) solution. The CCV solution may be prepared by mixing 2.0 mL of A(if the more concentrated solution is used) and 2.0 mL of B(if the more concentrated solution is used) and diluting to 100 mL with eluent. The LCS solution may be prepared by mixing 10 mL of A and 10 mL of B and diluting to 100 mL with eluent.

LCS & Continuing Calibration Verification Solution Analyte	Final Conc. (V_f=5ml)
Fluoride	2.5 mg/L
Chloride	50. mg/L
Nitrite	2.5 mg/L
Bromide	10. mg/L
Nitrate	2.5 mg/L
Orthophosphate	2.5 mg/L
Sulfate	50. mg/L

7.7.3 Prepare or purchase a primary stock standard(s). Dilute these stock standards to prepare the mixture to be used for the Matrix Spike solution. Alternatively purchase these mixes (ready to use) from a commercial source. Add 100 uL of each IC Spike solution (the more concentrated)to 5 mL of sample when preparing the MS. Dilute as needed after spiking the sample.

Matrix Spike "True" Values

Analyte	Final Conc.
Fluoride	2.5 mg/L
Chloride	50. mg/L
Nitrite	2.5 mg/L
Bromide	10. mg/L
Nitrate	2.5 mg/L
Orthophosphate	2.5 mg/L
Sulfate	50. mg/L

Note: Stock standards, calibration standard #9 and LCS standard should be stored in the dark at 4° ? 2°C. Replace these standards when instrument response indicates target analyte degradation may have occurred or after the standard has expired (12 months commercial mix or six months in-house mix),

whichever occurs first. Nitrite and ortho-phosphate are particularly light and oxygen-sensitive.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to ensure a representative sample; allow for replicate analysis, if required; and minimize waste disposal.

8.2 Sample preservation and holding times for the anions that can be determined by this method for water samples are as follows:

Analyte	Preservation	Holding Time
Fluoride	4° ? 2°C	28 days
Chloride	4° ? 2°C	28 days
Nitrite	4° ? 2°C	48 hours
Bromide	4° ? 2°C	28 days
Nitrate	4° ? 2°C	48 hours
Orthophosphate	4° ? 2°C	48 hours
Sulfate	4° ? 2°C	28 days

Note: Soil leachates will follow the same preservation and holding times as the water samples, starting from the time of extraction.

9. QUALITY CONTROL

9.1 The TestAmerica Quality Control (QC) Program document provides further details of the QC and corrective action guidelines presented in this SOP. Refer to this document if additional guidance is required.

9.2 Initial Demonstration of Capability

9.3.1. Prior to the analysis of any samples by ion chromatography, the following requirements must be meet:

9.3.1.1. Method Detection Limit (MDL): An MDL must be determined prior to analysis of any samples. The MDL is determined using seven replicates of eluent spiked with the anions of interest that has been carried through the entire analytical procedure. See Section 9.11 for additional information on MDLs.

9.4 Linear Calibration Range (LCR)

9.4.1 The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the

resulting curve is linear. The verification of linearity must use a minimum of three standards. If any verification data exceeds the initial values by ±10%, linearity must be re-established. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

9.4.2 Batch definition: Preparation and QC batch definitions are provided in the TestAmerica QC Policy.

9.5 Method Blank (MB) or Laboratory Reagent Blank (LRB):

9.5.1 One MB must be processed with each preparation batch. The MB consists of eluent or deionized water that has been taken through the entire preparation and analytical process. For one-hour leach samples, the MB consists of 10 g of cleaned Ottawa sand with 100 mL of eluent or deionized water. The MB must go through the entire leaching process with the samples. The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The MB should not contain any analyte of interest above the reporting limit.

9.5.2 Corrective Action for MBs

9.5.2.1 If the analyte level in the MB exceeds the reporting limit for the analytes of interest in the sample, all associated samples are re-prepared and re-analyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.5.2.2 If there is no analyte greater than the RL in the samples associated with an unacceptable MB, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.6 Laboratory Control Sample (LCS) or Laboratory Fortified Blank (LFB):

9.6.1 One LCS must be processed with each preparation batch and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. If the result is outside established control limits the system is out of control and corrective action must occur. A control limit of 90 – 110% recovery must be applied. Corrective action will be re-preparation and reanalysis of the batch. The LCS is prepared from a separate stock standard, or neat material, of a different manufacturer than the stock, or neat material, used to prepare the calibration standard. For one-hour leach samples, the LCS consists of 10 g of cleaned Ottawa sand and 100 mL of LCS prepared solution. The LCS must go through the entire leaching process with the samples.

9.7.1 Corrective Action for LCS

9.7.1.1 If any analyte is outside established control limits, the system is out of control and corrective action must occur.

9.7.1.2 Corrective action will be re-preparation and re-analysis of the batch unless the client agrees that other corrective action is acceptable.

9.7.1.3 The only exception is if the LCS recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

9.8 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.8.1 One MS/MSD pair must be analyzed every ten samples. A matrix spike (MS) is a field sample to which a known concentration of target analyte has been added. Some client-specific DQOs may require the use of sample duplicates in place of or in addition to MS. The MS result is used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Spiking levels will be the same as the LCS values.

9.8.1.1 If the MS/MSD recovery or relative percent difference (RPD) falls outside the acceptance range, the recovery of the analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 80-120% recovery and 20% RPD must be applied to the MS/MSD.

9.8.1.2 If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is automatically flagged with a "4" flag. . T

9.8.1.3 If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted.

9.8.1.4 If the recovery of the LCS is outside the limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.9 Continuing Calibration Verification/Continuing Calibration Blank (CCV/CCB):

9.9.1 Continuing calibration is verified by analyzing the calibration standard after every ten samples. The CCV must fall within ? 10% of the true value for each target analyte. A CCB is analyzed immediately following the CCV to monitor low level accuracy and system cleanliness. The CCB result must be below the reporting limit for that analyte. If either the CCV or CCB fail to meet criteria, the analysis must be terminated, the problem corrected and re-preparation and analysis of all samples following the last CCV and CCB which were in control.

9.10 Control Limits

9.10.1 Control limits are specified in the method.

9.10.2 Control limits are easily accessible via LIMs

9.11 Method Detection Limits (MDLs) and MDL Checks

9.11.1 MDLs and MDL Checks are established by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.11.2 MDLs are easily accessible via LIMs

9.12 Nonconformance and Corrective Action

9.12.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action.

10. CALIBRATION AND STANDARDIZATION

10.1. Establish ion chromatographic operating parameters equivalent to those indicated in Table 1. Refer to Table 2 for typical standard run retention times. Other than the presence of the analytical column, the instrument conditions are the same.

10.2. For each analyte of interest, prepare a **minimum** of three calibration standards by adding accurately measured volumes of one or more stock standards to a volumetric flask and dilution to volume with eluent. If a sample analyte concentration exceeds the calibration range the sample may be diluted to fall within the range. If this is not possible, then three new calibration concentrations must be chosen—two of which must bracket the concentration of the sample analyte of interest. Each attenuation range of the instrument used to analyze a sample must be calibrated individually.

10.3. Using an injection volume of 25 µL of each calibration standard, tabulate peak height or area responses against the concentration. The results are used to prepare a calibration curve for each analyte. During this procedure, retention times must be recorded. All analytes will be calibrated using a least squares-linear regression, weighted least squares, quadratic regression or average response that is not forced through the origin. Correlation coefficients (R^2) must be 0.995 or better. An average response criterion is ± 20%.

10.4. Initial Calibration Verification (ICV) – The Initial Calibration is verified immediately following calibration prior to sample analysis. The acceptance criterion is ±10% of the true value. If this criterion is not met, the instrument must be re-calibrated.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo.

11.2. Any unauthorized deviations from this procedure must also be documented as a

nonconformance with a cause and corrective action described.

11.3. Table 1 summarizes the recommended operating conditions for the DX-120 and DX-320 ion chromatographs. Included in table 2 are estimated retention times that can be achieved by this method. Other columns, chromatographic conditions, or detectors may be used if the requirements of Section 9.2 are met.

11.4. Table 3 summarizes the recommended operating conditions for the ICS-2100 ion chromatograph. Included in table 4 are estimated retention times that can be achieved by this method for this instrument.

11.5. Check system calibration daily as outlined in table 3; and, if required, recalibrate as described in Section 10.

11.6. Prescreening may be performed using a conductivity meter to determine dilutions for chloride and sulfate. Follow this formula to approximate required dilutions:

$$\frac{Conductivity}{400} = \textbf{DILUTION FACTOR}$$

11.7. Load and inject a fixed amount (25 ?L) of sample. If the sample is cloudy then it should be filtered prior to loading into the autosampler. Flush injection loop thoroughly, using each new sample. Use the same size loop for standards and samples. Record the resulting peak size in area or peak height units. An automated constant volume injection system may also be used.

11.8. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of various concentrations. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms since retention time is concentration dependent for most analytes.

11.9. If the response for the peak exceeds the working range of the system, dilute the sample with an appropriate amount of eluent or deionized water and re-analyze.

11.10. If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and re-analyze.

Note: Retention time is affected by concentration. Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. See Table 2. In some cases, this peak migration may produce poor resolution or identification.

11.11. The following extraction should be used for solid materials. Add an amount of reagent water equal to ten times the weight of dry solid material taken as a sample. This slurry is

mixed for one hour using a magnetic stirring device or tumbler. Filter the resulting slurry before injecting using a 0.45 ? m membrane type filter. This can be the type that attaches directly to the end of the syringe

11.12. Should more complete resolution be needed between peaks the eluent (Section 7.3) can be diluted. This will spread out the run but will also cause the later eluting anions to be retained longer. The analyst must determine to what extent the eluent is diluted. This dilution should not be considered a deviation from the method.

12. ANALYTICAL DOCUMENTATION

12.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

12.2. Record all standards and reagents in the LIMS reagents module. All standards and reagents are assigned a unique number for identification.

12.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

12.4. Record all sample results and associated QC in LIMS. Level I and level II review are performed in LIMS.

13. DATA ANALYSIS AND CALCULATIONS

13.1. Prepare a calibration curve for each analyte by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve. Multiply answer by appropriate dilution factor. The quadratic curve uses the following functions:

$$y = ax + cx^2 + b$$

Where c is the curvature

y = Instrument response
x = Concentration
a = Slope
b = Intercept

13.2. Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and re-analyzed. Estimated values may be reported by client request. The following equations are used to calculation sample concentrations.

13.3. Aqueous and Non-Aqueous samples

Final Result (Mg/L or Mg/Kg) = (C) (D)

Where:

C=Concentration from Calibration Curve

D=Dilution Factor (If Needed)

13.4. Report results in mg/l for aqueous samples and mg/kg for one-hour leachates and mg/l for 18-hour leachates of solid samples.

13.5. Report $NO2^-$ as N, NO_3^- as N, $HPO_4^=$ as P

14. METHOD PERFORMANCE

14.1. The reporting limits for the following analytes are based on a 25 ul injection volume:

Analyte	Water RL (mg/L)	Soil RL (mg/kg)
Fluoride	1.0	10
Chloride	1.0	10
Nitrite	0.1	5
Bromide	0.5	5
Nitrate	0.1	5
O-Phosphate	0.5	5
Sulfate	1.0	10

14.2. The group/team leader has the responsibility to ensure this procedure is performed by an analyst who has been properly trained in its use and has the required experience. The group/team leader must document the training, and submit the results to the QA dept. for inclusion in the associate's training files.

15. POLLUTION PREVENTION

15.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in section 13 of the corporate environmental health and safety manual (cw-e-m-001) for "waste management and pollution prevention".

16. WASTE MANAGEMENT

16.1. All waste will be disposed of in accordance with Federal, State and Local regulations.

Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

16.2. Waste Streams Produced by the Method

16.3. The following waste streams are produced when this method is carried out.

16.3.1. Spent samples: Solid samples are disposed of as solid debris waste in the container labeled "Solid Waste."

16.3.2. Alkaline and/or acidic waste generated by the analysis. Aqueous waste can be poured down the drain if the pH is between 5 and 10. Any sample waste generated that is not in this pH range must be collected and disposed of in the designated acid waste drum located in the lab. This waste is collected in the laboratory in a designated container identified as "Acid Waste".

16.3.3. Contaminated plastic materials such as IC syringes, filters, caps and vials utilized for sample preparation. This waste is disposed of in containers labeled "Solid Waste."

17. REFERENCES

17.1. References

17.1.1. Method 300.0A, "Determination of Inorganic Anions by Ion Chromatography", Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio, Revision 2.1, August 1993

17.1.2. Method 9056A, "Determination of Inorganic Anions by Ion Chromatography", SW846, Test Methods for Evaluating Solid Waste, Third Edition, Draft Revision 1, September 1999

17.1.3. TestAmerica Canton Quality Assurance Manual (QAM), current version

17.1.4. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

17.1.5. Corporate Quality Management Plan (CQMP), current version

17.1.6. Revision History

Historical File:		Revision 3: 10/03/00		Revision 10: 10/20/10
		Revision 4: 11/06/04		Revision 11: 1/10/12
		Revision 5: 05/26/06		
		Revision 6: 06/27/08		
		Revision 7: 09/15/09		
		Revision 8: 04/29/10		
		Revision 9: 08/16/10		

18. ASSOCIATED SOPS AND POLICIES

18.1. QA Policy, QA-003

18.2. Glassware Washing, NC-QA-014

18.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

18.4. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

18.5. Supplemental Practices for DoD Project Work, NC-QA-016

18.6. Standards and Reagents SOP, NC-QA-017

18.7. Selection of Calibration Points, CA-T-P-002

18.8. Calibration Curves (General), CA-Q-S-005

18.9. Acceptable Manual Integration Practices, CA-Q-S-002

19. METHOD DEVIATIONS

19.1. The CCV and Laboratory Control Sample (LCS) solutions are made fresh weekly

19.2. Due to limitations in the data acquisition system, no zero point is run in the calibrations. The Linear Calibration Range will be determined using three standards.

20. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

20.1. Reporting limits

20.1.1 If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

20.2 The lab defaults to holding times from Method 300.0.

20.3 All working standards are prepared weekly.

20.4 Attachment 1 – Method Flow Chart

20.5 Tables

20.5.1 Table 1 – Standard Instrument Operating Parameters for DX-120 and DX-320

20.5.2 Table 2 – Retention Time Matrix for DX-120 and DX-320

20.5.3 Table 3 - Standard Instrument Operating Parameters for ICS-2100

20.5.4 Table 4 - Retention Time Matrix for ICS-2100

20.6 Figures

20.6.1 Figure 1 - Example Ion Chromatogram- DX-120 and DX-320

20.6.2 Figure 2 - Example Ion Chromatogram– ICS 2100

ATTACHMENT 1

Determination of Inorganic Anions by Ion Chromatography

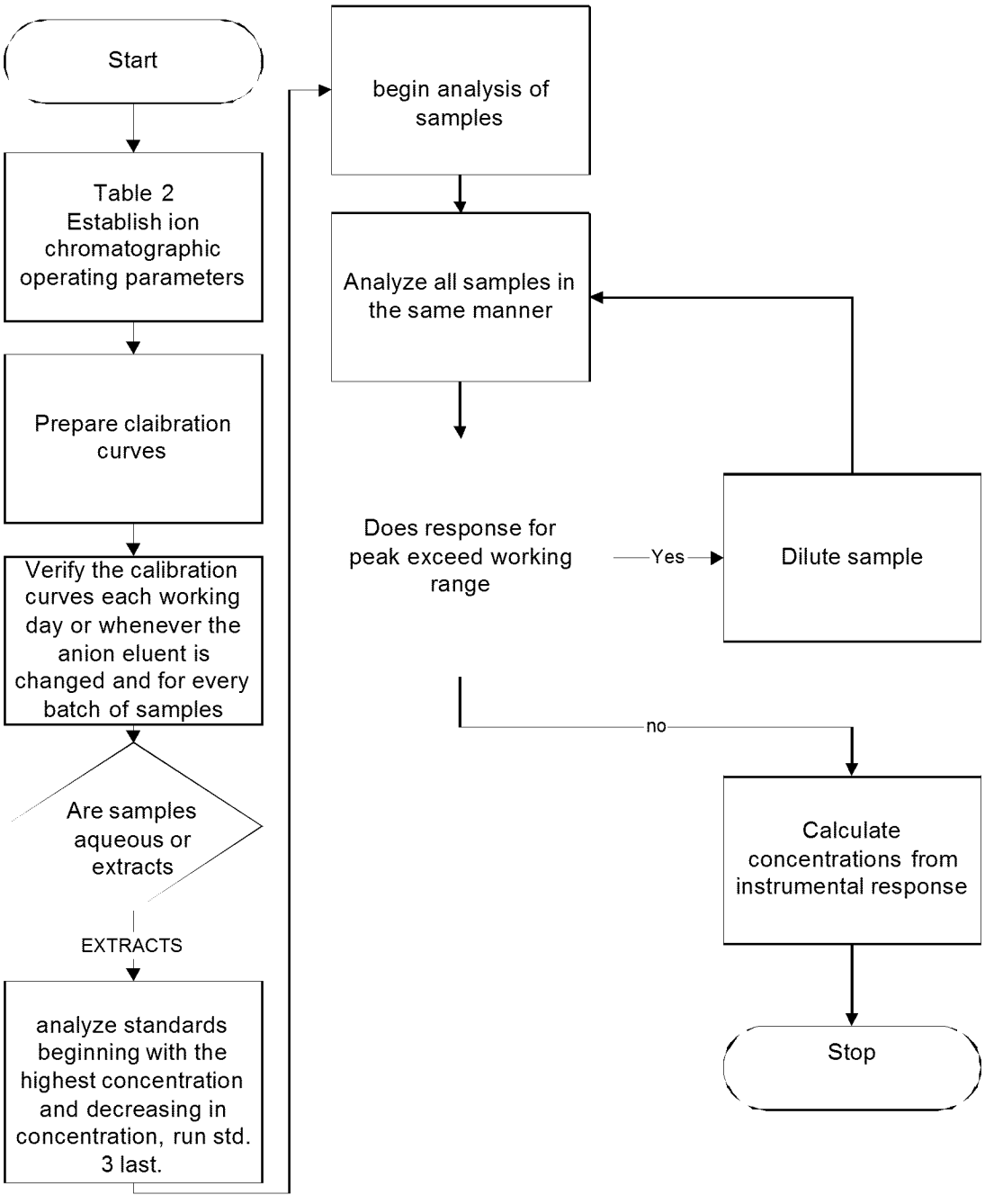


TABLE 1

Standard Instrument Operating Parameters

Standard Conditions:

Eluent Pump Rate:	1.20 mL/min (DX-120 and DX-320)
Sample Loop:	25 uL
Eluent:	1.0mM sodium bicarbonate, 3.5mM sodium carbonate
Detector Output:	Baseline conductivity should be 15 - 20 uS prior to sample analysis

TABLE 2

Standard Run Retention Time Matrix (minutes)*

DX-120 and DX-320

	Concentration (mg/L)												
Analyte	**0.05**	**0.2**	**0.5**	**1**	**2**	**2.5**	**5**	**10**	**20**	**40**	**50**	**100**	**200**
F^-	2.75		2.75			2.75	2.75	2.75					
Cl^-				3.97				3.98			4.03	4.08	4.17
NO_2^-	4.80		4.80			4.78	4.78	4.80					
Br^-		6.15			6.13			6.10	6.08	6.07			
NO_3^-	7.33		7.27			7.17	7.13	7.07					
$o\text{-}PO_4^{2-}$	9.53		9.53			9.52	9.50	9.48					
SO_4^{2-}				11.50				11.48			11.43	11.38	11.27

* Analyte retention time is concentration dependent for most anions. Retention time increases with increasing concentration for chloride. Retention time decreases with increasing concentration for bromide, nitrate, ortho-phosphate, and sulfate.

TABLE 3

Standard Instrument Operating Parameters

Standard Conditions:

Eluent Pump Rate: 1.00 mL/min (ICS-2100)

Sample Loop: 25 uL

Eluent: Varies due to gradient used during analysis, Startup concentration is 20mM

Detector Output: Baseline conductivity should be >1 uS prior to sample analysis

TABLE 4

Standard Run Retention Time Matrix (minutes)*

ICS-2100

Analyte	Concentration (mg/L)												
	0.05	0.2	0.5	1	2	2.5	5	10	20	40	50	100	200
F^-	3.98		3.98			3.98	3.99	3.99					
Cl^-				6.20				6.217			6.21	6.22	6.24
NO_2^-	7.49		7.50			7.49	7.48	7.47					
Br^-		9.14			9.14			9.12	9.10	9.08			
NO_3^-	10.18		10.17			10.15	10.12	10.07					
$o\text{-}PO_4^{2-}$	15.20		15.20			15.16	15.14	15.09					
SO_4^{2-}				11.20				11.19			11.08	11.00	10.86

FIGURE 1

EXAMPLE ION CHROMATOGRAM

DX-120 and DX-320

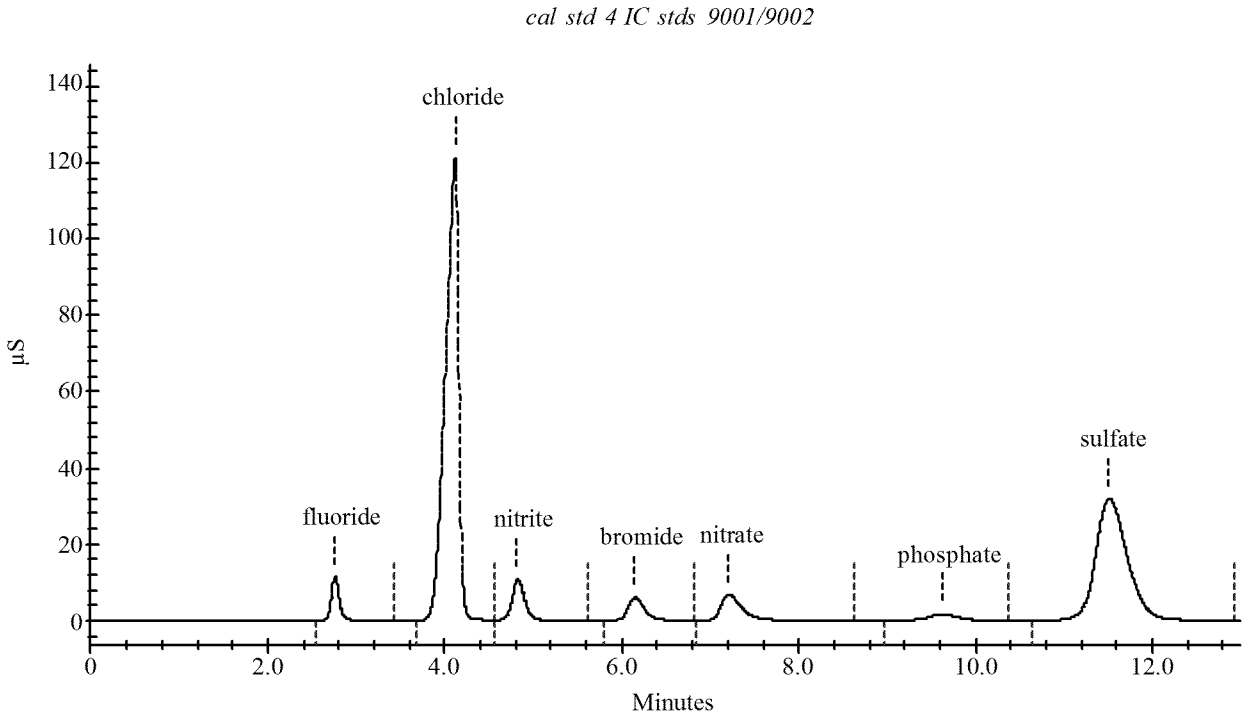
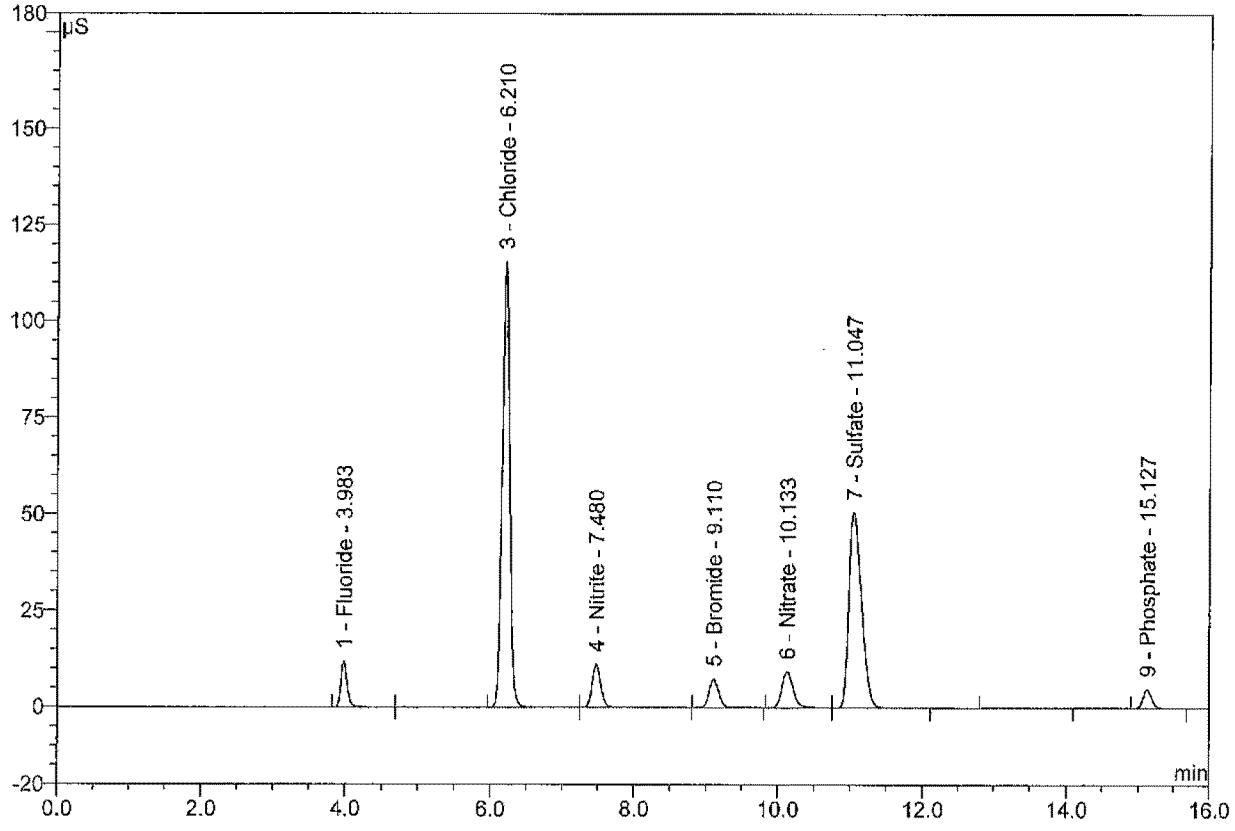


FIGURE 2

EXAMPLE ION CHROMATOGRAM

ICS-2100



TestAmerica Canton

SOP Amendment Form

SOP NUMBER: NC-WC-017 Rev. 3 SOP Effective Date: 7/25/13

SOP TITLE: Total Organic Carbon (TOC)

REASON FOR ADDITION OR CHANGE: Addition noted below

CHANGE EFFECTIVE FROM: (DATE): 8/23/13

Change(s) Made: Added the following section(s) to the SOP:

10.2. Initial Calibration Verification/Initial Calibration Blank (ICV/ICB)

10.2.1. The calibration is verified by using a midrange ICV from a second source. The ICV must not vary from the original curve by more than ± 10%, or recalibration is required. An ICB sample is analyzed after the ICV. It cannot contain the analyte of interest above the reporting limit, or recalibration is required.

EDITED BY/DATE: Melissa Fuller-Gustavel 8/22/13

*APPROVED BY:

Lucas Grossman

Technical Reviewer Signature Date 8/22/13

Rebecca Strait

QA Manager Signature Date 8/22/13

Laboratory Director Signature Date 8/24/13

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Canton

SOP No. NC-WC-017, Rev. 3
Effective Date: 7/25/13

Title: TOTAL ORGANIC CARBON (TOC)

[Method: SW846 Method 9060, 9060A, EPA Method 415.1, and SM 5310C]

Approvals (Signature/Date):

[redacted]	07/24/13	[redacted]	07/24/13
Technology Specialist	Date	Health & Safety Coordinator	Date
[redacted]	07/24/13	[redacted]	07/22/13
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. NC-WC-017, Rev 2.7, dated 10/22/10

Facility Distribution No. _____ **Distributed To: _____**

TABLE OF CONTENTS

1. SCOPE AND APPLICATION

1.1 This method is applicable to the determination of Total Organic Carbon (TOC) in waters and similar matrices. It is based on SW846 Methods 9060 and 9060A, EPA Method 415.1, and SM 5310C. The working linear range is instrument dependent at 1 mg/L to 50 mg/L with a reporting limit of 1 mg/L.

1.2 This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1 Organic carbon is measured using a carbonaceous analyzer. This instrument converts the organic carbon in a sample to carbon dioxide (CO_2) by wet chemical oxidation. The CO_2 formed is then measured by nondispersive infrared (NDIR) detector. The amount of CO_2 in a sample is directly proportional to the concentration of carbonaceous material in the sample.

3. DEFINITIONS

3.1 Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1 Contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts may cause Method interferences. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2 Carbonate and bicarbonate interfere but are eliminated by the acidification and purging step of the instrument. Note that the removal of carbonate and bicarbonate by acidification and purging with nitrogen, or other inert gas, can result in the loss of volatile organic substances.

4.3 This procedure is applicable only to homogeneous samples, which can be injected into the apparatus reproducibly by means of a Microliter-type syringe or pipette. The openings of the syringe or pipette limit the maximum size of particle, which may be included in the sample.

4.4 Large organic particles or very large or complex organic molecules such as tannins, lignins, and humic acid may be oxidized slowly because persulfate oxidation is rate-limited.

4.5 Persulfate oxidation of organic molecules is slowed in samples containing significant concentrations of chloride above 0.05%. To remove this interference, extend reaction time and/or increase amount of persulfate solution in heated-persulfate instruments.

5. SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2 The auto-sampler has a probe that is sharp. Use caution not to stick yourself.

5.3 The furnace is very hot and can cause severe burns if touched.

5.4 Sodium persulfate is a strong oxidizer. Avoid contact with combustible materials, organic materials, strong reducing agents, and excess heat.

5.5 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Phosphoric Acid	Corrosive	1 mg/m^3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.

Sodium Persulfate	Oxidizer Corrosive	0.1 mg/m^3-TWA as Persulfates	Causes irritation to the respiratory tract. Symptoms may include sore throat, shortness of breath, inflammation of nasal passages, coughing, and wheezing. Causes severe irritation or burns to the skin and eyes. Symptoms include redness, itching, pain and burns. May cause allergic skin reactions. Can cause eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.6 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.7 Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.8 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.9 It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.10 Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.11 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1 O·I Corporation Model 1010 TOC Analyzer with 1051 vial multi-sampler

6.2 Nitrogen Gas and Regulator

6.3 Volumetric flasks: Various sizes

6.4 Volumetric pipettes: Various sizes

6.5 Vials: 40 mL glass

6.6 Graduated cylinders: Various sizes

6.7 0.45 micron filters (for DOC analysis only)

6.8 Whatman filter #4

6.9 Top loading balance: Capable of accurately weighing ± 0.01 g

7. REAGENTS AND STANDARDS

7.1. All standards and reagents are stored according to manufacturer's specification unless otherwise noted in this SOP.

7.2. Reagents

7.2.1. Sodium Persulfate: Reagent Grade

7.2.2. Sodium Persulfate Solution: Add 200 g sodium persulfate ($Na_2S_2O_8$) to a 1 liter volumetric flask and dilute to volume with reagent water. Remake after 6 months or as needed.

7.2.3. Phosphoric Acid, concentrated: Reagent Grade

7.2.4. Phosphoric Acid Solution: Carefully add 59 mL concentrated phosphoric acid (H_2PO_4) to 900 mL of reagent water in a 1 liter volumetric flask. Dilute to volume with reagent water. Remake after 1 year or as needed.

7.2.5. Sulfuric Acid, concentrated: Reagent Grade

7.3. Standards

7.3.1. All standards should be prepared in volumetric flasks using volumetric pipettes, and diluted to volume with reagent water.

7.3.2. TOC Stock Standard

7.3.3. Primary and secondary sources are needed.

7.3.3.1. TOC 1000 mg/L

7.3.3.1.1. Dilute 1.06 g KHP (potassium acid phthalate) to volume in a 500 mL volumetric flask. A commercially prepared solution may also be used.

7.3.3.1.2. Prepare every six months.

7.3.4. Inorganic Carbon (TIC) Stock Solution

7.3.4.1. Carbonate-Bicarbonatestock solution: 1000 mg/L carbon: Weigh 0.3500 g of sodium bicarbonate and 0.4418 g of sodium carbonate and transfer both to the same 100 mL volumetric flask. Bring to volume with DI. Make each day of use.

7.3.4.2. Carbonate-Bicarbonatestandard solution, 50 mg/L: Pipette 5 mL of the 1000 mg/L stock solution into approximately50 mL of DI in a 100 mL volumetric flask. Dilute to volume with DI. Make each day of use.

7.3.5. TOC Calibration Standards

7.3.6. Prepare the following standards from the primary stock standard described in Section 7.3.3.1.1.

Concentration (mg/L)	Volume (mL)	Stock Concentration (mg/L)	Final Volume (mL)
50	5	1000	100
25	2.5	1000	100
10	1	1000	100
5	0.5	1000	100
0.5	0.05	1000	100

7.3.7. Prepare the following standards from the secondary stock standard described in Section 7.3.3.1.1.

Concentration (mg/L)	Volume (mL)	Stock Concentration (mg/L)	Final Volume (mL)
25 (CCV/MS/MSD)	6.25	1000	250

7.3.8. TOC Verification Standard (LCS)

7.3.8.1. A commercially prepared solution is used.

7.3.9. Inorganic Verification Standard

7.3.9.1. Annually, or after major instrument maintenance, check efficiency of inorganic carbon removal by analyzing the 50 mg/L inorganic standard solution (section 7.2.4.2).The TOC result must be less than the RL.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Samples are preserved to a pH <2 with sulfuric acid (H_2SO_4) or hydrochloric acid (HCl) and stored in plastic or glass vials or bottles protected from sunlight and atmospheric oxygen at 4°C±2°C.

8.2. The holding time is 28 days from sampling to analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD), which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank (MB)

9.2.1. One MB must be processed with each preparation batch. The MB consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The MB should not contain any analyte of interest at or above the reporting limit.

9.2.2. A MB consisting of 40 mL of reagent water and all reagents added to the samples must be prepared and analyzed with each batch of samples. The MB is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data.

9.2.3. For Method 5310C, the MB must be run in replicate, and the average of the two runs reported.

9.2.4. Corrective Action for MBs

9.2.4.1. If the analyte level in the MB exceeds the reporting limit for the analytes of interest in the sample, all associated samples are re-prepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.4.2. If there is no analyte greater than the RL in the samples associated with an unacceptable MB, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. LCSs are well characterized, laboratory generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS is used to monitor the accuracy of the analytical process, independent of matrix effects. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.3.2. A purchased LCS must be analyzed with each batch of samples.

9.3.3. For Method 5310C, the LCS must be run in replicate, and the average of the two runs reported.

9.3.4. Corrective Action for LCS

9.3.4.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.4.2. The only exception is that if the LCS recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

9.3.4.3. Corrective action will be repreparation and re-analysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix

spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. An MS/MSD consisting of 20 mL of sample and 20 mL of the 25 mg/L standard will be analyzed with each analytical batch of samples.

NOTE: For Method 9060, one MS/MSD pair is required per 10 samples analyzed.

NOTE: For Method 5310C, the MS/MSD must be run in replicate, and the average of the two runs reported.

9.4.3. Corrective Action for MS/MSDs

9.4.3.1. If the analyte recovery or Relative Percent Difference (RPD) falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and re-analysis of the batch.

9.4.3.2. If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data is reported and flagged with a "4" in LIMS.

9.5. Control Limits

9.5.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.5.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMs.

9.6. Method Detection Limits (MDLs) and MDL Checks

9.6.1. MDLs and MDL checks are established by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.6.2. MDLs are easily accessible via LIMs.

9.7. Nonconformance and Corrective Action

9.7.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action.

10. CALIBRATION AND STANDARDIZATION

10.1. Recommended Initial Setup

Constant Settings		
STD Mass	=	6.76 ug C
Sample Vol	=	2.0 mL
Acid Vol	=	4 x 100 uL
Oxidant Vol	=	10 x 100 uL

10.1.1. Adjust the nitrogen to 60 $\pm$ 10 psi using the flow valve on the tank.

10.1.2. Remove the reagent bottles and fill with appropriate reagents (phosphoric acid solution and sodium persulfate). Do not fill bottles completely full; leave a small amount of air space. Loosely reconnect caps (and tubing), and replace the bottles into the instrument.

10.1.3. Blank and calibrate the instrument when CCVs and/or CCBs fail to meet acceptance criteria or when other problems are encountered.

10.1.3.1. Choose "Calibration". Start a new file with the current date.

10.1.3.2. Choose "Sequences" from the "databases" menu option, open up the calibration template.

10.1.3.3. Confirm all information. If blanking is required, it is best if done before calibration. Enter the desired number of blanks (no less than five) in the "reagent blanks before" field.

10.1.3.4. Analyze an ICV/ICB

10.1.3.5. Save the file using the current date as the filename.

10.1.3.6. Update data file information.

10.1.3.6.1. Choose the setup menu option, go into win TOC output, change the log fill name and prefix counter.

10.1.3.7. When analysis is complete, print the run from "/utilities/view run log".

10.1.3.8. Evaluate the data. The correlation coefficient of the original curve must be =0.995 or recalibration is required.

10.2. Initial Calibration Verification/Initial Calibration Blank (ICV/ICB)

10.2.1. The calibration is verified by using a midrange ICV from a second source. The ICV must not vary from the original curve by more than ± 10%, or recalibration is required. And ICB sample is analyzed after the ICV. It cannot contain the analyte of interest above the reporting limit, or recalibration is required.

10.3. Continuing Calibration Verification (CCV)

10.3.1. The run is checked at the beginning, after every ten samples, and at the end of the run of the same species using a midrange CCV made from a secondary source (Section 7.2.4. or 7.2.7.) to verify continued linearity. A CCV cannot vary from the original curve by more than ± 10%, or re-analysis of samples bracketed by the failing CCV is required. Recalibration may be necessary if problem persists.

10.3.2. CCVs that bracket samples being analyzed by Method 5310C must be run in replicate.

10.3.3. System cleanliness is checked every ten samples and at the end of the run using Continuing Calibration Blank (CCB). A CCB cannot contain the analyte of interest above the reporting limit, or re-analysis of samples bracketed by the failing CCB is required. Recalibration may be necessary if problem persists.

10.3.3.1. CCBs that bracket samples being analyzed by Method 5310C must be run in replicate.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo.

11.2. Sample Preparation Procedure

11.2.1. If excess particulate matter exists, filter an aliquot of sample through a Whatman #4 filter into a TOC vial or decant. Narrate the data accordingly.

11.2.2. If DOC analysis is requested, and samples are not filtered in the field, an unpreserved sample aliquot will be filtered using a 0.45 micron filter.

11.2.2.1. A filtered blank must be analyzed with DOC samples requiring lab filtration.

11.3. Sample Analysis Procedure

10.3.1 Type a run protocol sequence into the computer using the run template, if desired. Update the data file information in the setup/win TOC output.

10.3.2 For Method 9060, quadruplicate analysis is required on samples. If quadruplicate reporting is requested, each of four results is reported. Replicate analysis should be taken from separate vials, if available. If only one reportable result is requested per sample, the four results should be taken from one vial, and the average of the four results are reported.

10.3.3 For Method 5310C, replicate analysis is required for samples ***and standards***. Two results should be taken from one vial, and the average of the two results is reported. Both results will be reported upon client request

10.3.4 For Method 415.1, only one analysis is required. The single analysis is reported directly from the instrument printout.

10.3.5 All samples and standards should be poured into 40 mL vials. Samples received in vials can be run in those containers, provided there is not an excess of solids.

10.3.6 Be sure the samples are loaded on the sampler, the first one positioned under the needle.

10.3.7 Click the "Start" button.

10.3.8 Samples that fall outside the linear range (>50 mg/L) of the instrument must be diluted and reanalyzed.

10.3.8.1 Samples following a high sample should be re-analyzed if carryover is a concern.

10.3.9 Print the run from "utilities/view run log".

10.3.10 When analysis is complete, properly dispose of or put away samples and standards.

12. DATA ANALYSIS AND CALCULATIONS

12.1 Preparation Documentation

12.1.1 Record any sample preparation in LIMS.

12.2 Analytical Documentation

12.2.1 Record all analytical information in LIMS, including any corrective actions or modifications to the method.

12.2.2 Record all standards and reagents in the LIMS Reagents module. All standards and reagents are assigned a unique number for identification.

12.2.3 Documentation, such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs, is available for each data file.

12.2.4 Record or upload all sample results and associated QC into LIMS. Level I and Level II review is performed in LIMS

12.2.5 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12.3 Calculations

12.3.1 *LCS % Recovery* = $\dfrac{\textit{Instrument values } *}{\textit{True Value}} x\ 100$

12.3.2 MS/MSD % recovery =

$$\left(\frac{\textit{(Instrument values * MS or MSD) - (Avg sample instrument value * \div 2)}}{\textit{12.5}}\right) x100$$

* For 9060 and 9060A analysis, one of the values may be judged erroneous and disregarded if three of the four are consistent. If no consistency can be found in the four values, the sample must be rerun.

13. METHOD PERFORMANCE

13.1 Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2 . Training Qualifications

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2 Waste Streams Produced by This Method:

15.2.1 The following waste streams are produced when this method is carried out.

15.2.1.1 Acidic waste from the auto-analyzer: This waste is disposed of in a designated container identified as "Acid Waste."

15.2.2 Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by annual refresher training.

16. REFERENCES

16.2. References

16.2.1. SW846, Test Methods for Evaluating Waters, Third Edition, Total Organic Carbon, Method 9060.

16.2.2. SW846, Test Methods for Evaluating Waters, Total Organic Carbon Method 9060A, Rev. 1, November 2004.

16.2.3. EPA 600, Methods for Chemical Analysis of Water and Wastes, Organic Carbon, Method 415.1.

16.2.4. Standard Methods for the Examination of Water and Wastewater, Method 5310C, 2001

16.2.5. Corporate Quality Management Plan (CQMP), current version

16.2.6. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.2.7. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.2.8. Revision History

Historical File:		Revision 1: 11/20/96		Revision 2.4: 08/01/07
		Revision 2: 06/01/99		Revision 2.5: 04/30/08
		Revision 2.1: 08/17/00		Revision 2.6: 12/16/09
		Revision 2.2: 01/24/03		Revision 2.7: 10/22/10
		Revision 2.3: 12/16/04		

16.3. Associated SOPs and Policies, current version

16.3.1. QA Policy, QA-003

16.3.2. Glassware Washing, NC-QA-014

16.3.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.3.4. Method Detection Limits, NC-QA-021 and CA-Q-S-006

16.3.5. Supplemental Practices for DoD Project Work, NC-QA-016

16.3.6. Standards and Reagents, NC-QA-017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.2. Reporting Limits

17.2.1. The lower reporting limit is 1 mg/L

17.2.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.3. TroubleshootingGuide

17.3.1. See the manufacturer's instructions for an instrument troubleshooting guide and maintenance requirements.

17.4. Method Deviations

17.4.1. A blender is not used to homogenize samples.

TestAmerica Canton

SOP Amendment Form

SOP NUMBER: NC-WC-017 Rev. 3 SOP Effective Date: 7/25/13

SOP TITLE: Total Organic Carbon (TOC)

REASON FOR ADDITION OR CHANGE: Addition noted below

CHANGE EFFECTIVE FROM: (DATE): 8/23/13

Change(s) Made: Added the following section(s) to the SOP:

10.2. Initial Calibration Verification/Initial Calibration Blank (ICV/ICB)

10.2.1. The calibration is verified by using a midrange ICV from a second source. The ICV must not vary from the original curve by more than ± 10%, or recalibration is required. An ICB sample is analyzed after the ICV. It cannot contain the analyte of interest above the reporting limit, or recalibration is required.

EDITED BY/DATE: Melissa Fuller-Gustavel 8/22/13

*APPROVED BY:

Lucas Grossman

Technical Reviewer Signature Date 8/22/13

Rebecca Strait

QA Manager Signature Date 8/22/13

Laboratory Director Signature Date 8/24/13

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

CANTON

SOP No. NC-WC-010, Rev. 12
Effective Date: 03/21/13

Title: pH ELECTROMETRIC METHOD

[Method: SW846 Methods 9040B, 9040C, 9041A, 9045C and 9045D, EPA Method 150.1, SM4500 H^+B]

Approvals (Signature/Date):

Lucas Grossman 3/21/2013
Technology Specialist Date

3/21/2013
Health & Safety Coordinator Date

Rebecca Strait 3/21/2013
Quality Assurance Manager Date

3/21/2013
Laboratory Director Date

This SOP was previously identified as SOP NC-WC-010, Rev 11 dated 04/16/12

Facility Distribution No. _____ **Distributed To:_____**

TABLE OF CONTENTS

1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of pH in waters, drinking waters, wastewaters, and solids. It is based on SW846 Methods 9040B, 9040C. 9041A, 9045C, 9045D, SM4500 H^+B, and EPA Method 150.1. Method 9040B/9040C should be used if the aqueous phase constitutes at least 20% of the total volume of the waste. Method 9045C/9045D should be used for measuring pH in soils and waste samples. If water is present, it must constitute less than 20% of the total volume of the sample. See Section 11 for details on determining the correct method.

1.2. The approximate working range is 1 - 14 pH units.

1.3. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. The pH is determined electrometrically by using an electrode. The pH meter is calibrated with a series of known pH buffers.

2.2. For Method 9041A, an aliquot of sample is analyzed for pH using pH paper. Samples are mixed with water prior to analysis.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low sodium error electrode.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. There are no materials used in this method that have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

5.3. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.7. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.8. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported**immediately** to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. pH meter with electrode(s) and temperature compensation

6.2. Disposable beakers: various

6.3. Top loading balance: Capable of accurately weighing±0.01 g

6.4. Stir plate and stir bars

6.5. Shaker or mechanical tumbler

6.6. Autotitrator

6.7. Centrifuges tubes

6.8. pH paper: Various pH ranges

6.9. Disposable snap top containers

7. REAGENTS AND STANDARDS

7.1. Standards

7.1.1. A commercially available control standard (LCS).

7.1.2. Target Calibration Standards

7.1.2.1. pH 2, 4, 7, 10, and 12 buffers--purchased

Note: Buffers used for drinking water analysis must be discarded 6 months after opening.

7.1.2.2. Fresh buffers are poured and used each working day.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored in plastic or glass containers at 4°C ± 2°C.

8.3. Samples should be analyzed as soon as possible after sampling, but not to exceed one day after sampling.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS and Sample Duplicate) which are processed similarly with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Sample Duplicate

9.2.1. A sample duplicate (DU) is a second aliquot of an environmental sample, taken from the same sample container when possible, that is processed with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. The sample and DU results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample/DU precision results are not necessarily representative of the precision for other samples in the batch.

9.2.2. Sample duplicates are performed at a frequency of 10% per matrix, and must meet laboratory-specific limits for precision. For 9041A, all samples will be analyzed in duplicate.

9.3. Laboratory Control Sample (LCS)

9.3.1. One aqueous LCS must be processed with each analytical batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. A commercially available (Environmental Resource Associates or equivalent) control standard will be analyzed. Recovery must be within +/-3% of true value.

9.3.3. Corrective Action for LCS

9.3.3.1. If the pH is outside the established control limits the system is out of control and corrective action must occur.

9.3.3.2. Corrective action consists of identification and correction of the cause for the out of control situation and reanalysis of all effected samples.

10. CALIBRATION AND STANDARDIZATION

10.1. Initial Calibration

10.1.1. Refer to the manufacturer's manual for instrumental calibration.

10.1.2. Corrosivity analysis under 9040B and C require differing calibration buffers. Reference section 10.1.3 for pH, and section 10.1.4 for Corrosivity.

10.1.3. The following procedure is applicable for use with the Orion Star A211 pH meter.

10.1.3.1. One to five pH buffers can be used for calibrating this meter. If pH

buffers 2, 4,7,10 and 12 are used during the calibration samples can be reported for pH or corrosivity.

10.1.3.2. NOTE: For drinking water analysis, calibration buffers must be stirred during calibration.

10.1.3.3. Note: Methods 9040B and C require a calibration buffer of pH 2 and pH12 if corrosivity characterization is needed.

10.1.3.4. After calibration, run a pH 7, then an LCS, allowing the electrode to stabilize for each. Record results on analytical logsheet. See section 10.2.2 for additional information on continuing calibrations for corrosivity.

10.1.4. For 9040B and C Corrosivity, the Mettler Toledo S20 pH meter could be used as well for narrow range sample readings.

10.1.4.1. The S20 pH meter will require calibration dependant upon the sample's initial pH reading from the Orion Star A211.

10.1.4.2. Chose an appropriate 3 point calibration range (2, 4, and 7 or 7, 10, and 12) from the calibration menu.

10.1.4.2.1. Place the probe in the first calibrant and press "Cal".

10.1.4.2.2. Rinse the probe with DI water, place the probe in the second calibration buffer and press "Cal".

10.1.4.2.3. Repeat with the 3rd calibration buffer.

10.1.4.2.4.NOTE: Between every two calibration points, the instrument will define the offset and the slope. Manufacturer's recommended criteria for the offset is $\pm$ 15%, and slope criteria is 95 – 105. A three-point calibration will have two values for offset and slope. If either of these values fall outside of the manufacturer's criteria, recalibration is required.

10.1.5. The pH meter must be calibrated daily. The calibration date is recorded on the analytical logsheet.

10.1.6. If the pH meter has been turned off, it must be calibrated prior to use.

10.1.7. For drinking water analysis, the calibration slope must be 95 – 105 %, or recalibration must take place.

10.2. Continuing Calibration

10.2.1. For pH: A pH 7 buffer is analyzed before sample analysis, every ten samples, and at the end of the analysis to ensure the calibration remains linear.

10.2.2. For Corrosivity: Continuing Calibration level is dependant upon which level calibration curve is used. Samples analyzed for Corrosivity must be bracketed by the mid-point buffer from the calibration curve used for the sample. For example, a sample(s) with an initial value of 3.5 must be bracketed by passing pH 4 buffer checks if a 2, 4 and 7 calibration was performed.

10.2.3. The pH meter must be recalibrated if the buffer deviates by more than±0.05. If this range is exceeded, re-analyze all samples analyzed since the last pH buffer that met criteria.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo.

11.2. Sample Preparation

11.2.1. At the time of sample receipt, the sample must be inspected to determine the correct method reference. General Chemistry staff members will determine the percent of aqueous phase present and notify the Project Management staff if a method change is needed. . Methods 9040B and C should be used if the aqueous phase constitutes at least 20% of the total volume of the waste. Method 9045C and 9045D should be used for measuring pH in soils and waste samples. If water is present, it must constitute less than 20% of the total volume of the sample.

NOTE: The analyst will note in the comments section in LIMS which method was used and if a method change was required.

11.2.2. Waters and aqueous wastes where the aqueous phase constitutes at least 20% of the total volume of the waste

11.2.2.1. No preparation necessary for waters and wastewaters.

11.2.3. Solids and Sludges

11.2.3.1. Place 10 g (± 0.5 g) of sample in a beaker or other suitable container.

11.2.3.2. Add 10 mL of reagent water and mix for five minutes in a shaker or mechanical tumbler.

11.2.3.3. Allow sample to stand for about one hour to allow the solids to settle out.

11.2.4. Non-aqueous Waste and liquids

11.2.4.1. Place 10 g (± 0.5 g) of sample in a beaker or other suitable container.

11.2.4.2. Add 10 mL of reagent water and mix for five minutes in a shaker or mechanical tumbler.

11.2.4.3. Let the waste suspension stand for about 15 minutes to allow most the suspended waste to settle out from the suspension or filter or centrifuge off aqueous phase for pH measurement.
Note: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned if it becomes coated with an oily material.

11.2.5. Method 9041A Sample Preparation (solids, sludges, and oils)

11.2.5.1. Place 10 g (± 0.5 g) of sample in a snap cap.

11.2.5.2. Add 10 mL of reagent water to the sample and mix.

11.2.5.3. Allow the sample and water layers to separate and carefully decant the water layer into another snap cap for analysis. If it is not possible to decant without decanting some of the sample (in the case of oils or oily sludges), it is permissible to use a disposable transfer pipette to remove the water layer for analysis.

11.3. Sample Analysis

11.3.1. Manual Procedure

11.3.1.1. Waters and aqueous wastes where the aqueous phase constitutes at least 20% of the total volume of the waste

11.3.1.1.1. Place the sample in a clean beaker using a sufficient volume to cover the sensing elements of the electrode(s). Allow the pH to stabilize (swirling or stirring may quicken stabilization). Record the pH on the analytical logsheet. Remove the electrodes from the sample. Rinse and gently dab off the electrodes between each measurement. Store the electrodes in pH 7 buffer when not in use.

NOTE: If Corrosivity is requested, calibrate the Orion Star A211 using pH buffers 2,4,7,10 and 12 or Mettler Toledo S20 pH Meter according to the procedure listed in 10.1.4, and continue analyzing following steps 11.3.1.1.2 and 11.3.1.1.3 on that instrument.

11.3.1.1.2. For 9040B, 9040C, and 150.1 – Continuously stir the sample while obtaining a stable reading.

11.3.1.1.3 For Method 9040B, 9040C and 150.1 note and record the sample pH of the first aliquot. Repeat the measurement on successive aliquots of sample until the values differ by < 0.1 pH units. Two or three volume changes are usually sufficient. If more than three measurements are required, contact the Group Leader.

11.3.2. Solids and Non-aqueous Waste and liquids

11.3.2.1.Immerse the pH electrodes in the supernatant layer of the sample - be careful not to stir up solids. Allow pH to stabilize and record it on the analytical logsheet. Remove and rinse the electrodes between each measurement. Store electrodes in the pH 7 buffer.

NOTE: If the sample contains oil or other substances that will coat and damage the electrodes analyzing the pH by pH - Paper Method 9041A should be considered.

11.3.3. Automated Procedure-

11.3.3.1. Load the appropriate schedule on the autotitrator starting with the pH calibration.

11.3.3.2. Pour a homogenized sample into the centrifuge tubes and place the tubes in the appropriate position on the autosampler. Remember to include a pH 7 buffer check after every ten positions.

11.3.3.3. Start the autotitrator.

11.3.4. Method 9041A Sample Analysis Procedure

11.3.4.1. Immerse the wide range pH paper into the decanted water layer of the sample for several seconds. Remove the paper and determine the pH range from the manufacturer's pH chart. Using the appropriate narrow range pH paper, read the sample pH and record the pH on the analytical logsheet. Reading with the narrow range pH paper will be done in duplicate.

NOTE: The initial pH range check that is performed with the wide range pH paper does **not** count as a duplicate analysis.

11.4. Analytical Documentation

11.4.1. Record all analytical information in LIMS, including the analytical data from standards and any corrective actions or modifications to the method.

11.4.2. All standards are logged into the LIMS standards and reagents module. All standards are assigned a unique number for identification.

11.4.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs are scanned and attached to the Documents section in the LIMS analytical batch.

11.4.4. Sample results and associated QC are transferred directly into LIMS at the time of analysis. Level I and Level II review is done in LIMS.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not Applicable

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method

15.2.1. The following waste streams are produced when this method is carried out.

15.2.1.1. Acidic and alkaline sample waste and exhausted buffer solutions can be poured down the drain if the pH is between 5 and 10. Any sample waste generated that is not in this pH range is collected in a designated container identified as "Acid Waste".

15.2.1.2. Exhausted soil or oil samples analyzed by the method. The liquid layer is decanted and disposed of in a designated container identified as "Acid Waste". The remaining solid layer is disposed of by placing it in a container identified as "Solid Waste".

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica Canton. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks followed by annual refresher training.

16. REFERENCES

16.1. References

16.1.1. SW846, Test Methods for Evaluating Solid Waste, 3rd Edition, pH Electrometric Measurement, Method 9040B, Revision 2, January 1995

16.1.2. EPA 600, Methods for Chemical Analysis of Water and Wastes, pH (Electrometric), Method 150.1

16.1.3. SW846, Test Methods for Evaluating Solid Waste, 3rd Edition, Soil pH, Method 9045C, Revision 3, January 1995.

16.1.4. SW846, Test Methods for Evaluating Soil Waste, Soil and Waste pH Method 9045D, Revision 4, November 2004.

16.1.5. SW846, Test Methods for Evaluating Solid Waste, pH Electrometric Measurement , Method 9040C, Revision 3, November 2004.

16.1.6. SW846, Test Methods for Evaluating Solid Waste, Third Edition, pH paper, Method 9041A, Revision 1, July 1992

16.1.7. Standard Method for pH Electrometric Method, Eighteenth Edition, SM4500 H^+B

16.1.8. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.9. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan current version

16.1.10. Corporate Quality Management Plan (CQMP), current version

16.1.11. Ohio EPA Laboratory Manual for Chemical Analyses of Public Drinking Water 2000

16.1.12. Revision History

Historical File:		Revision 4.0: 01/04/9		Revision 10: 11/24/1(
		Revision 4.1: 11/28/00		Revision 11: 04/16/1
		Revision 5: 02/05/0		
		Revision 6: 10/27/0		
		Revision 7: 03/23/06		
		Revision 8: 04/30/0		
		Revision 9: 05/26/1(		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Supplemental Practices for DoD Project Work, NC-QA-016

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. A minimum reporting limit of 0.1 SU (standard units) is listed in LIMS. Units are reported as "SU".

17.2. Method Deviations

17.2.1. Method 9041A requires a procedure to identify interferences. The laboratory does not perform this procedure.

17.2.2. Method 9041A requires each batch of pH paper to be calibrated versus certified pH buffers or a pH meter which has been calibrated with certified pH buffers. The pH paper is not calibrated by the laboratory.

17.2.3. Method 9041A does not call for any kind of sample preparation; however, due to the various matrices encountered, the laboratory preps the samples as described in section 11.2.5.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Canton

SOP No. NC-WC-060, Rev. 8
Effective Date: 8/12/13

Title: SULFIDE

[Methods: SW846 Methods 9030B and 9034, EPA 376.1, and SM 4500-S2-F]

Approvals (Signature/Date):

Technology Specialist	Date	Health & Safety Coordinator	Date
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. NC-WC-060, Rev 7, dated 11/16/10

Facility Distribution No. _____ **Distributed To: _____**

TABLE OF CONTENTS

1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of the concentration of Sulfide in waters, liquids, solids, and sludges. It is based on SW846 Method 9030B/9034, Methods for Chemical Analysis of Water and Wastes EPA 376.1, and SM 4500-S2-F. For 9030B/9034 analysis, the working range is 3 to 30 mg/L for waters and 30-650 mg/kg for solids and sludges. Methods 376.1 and 4500 S2-F are suitable for measuring sulfide in concentrations above 1 mg/L.

1.2. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. For acid soluble sulfide samples, separation of sulfide from the sample matrix is accomplished by the addition of sulfuric acid to the sample. The sample is heated to 70^0C and the hydrogen sulfide (H_2S), which is formed, is distilled under acidic conditions and carried by a nitrogen stream into zinc acetate scrubbing bottles where it is precipitated as zinc sulfide.

2.2. An excess of iodine is added to a sample, which oxidizes the Sulfide to sulfur under acidic conditions. The excess iodine is back titrated with sodium thiosulfate.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), latest version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Reducing substances such as thiosulfite, sulfites, and various organic compounds cause interferences, but treatment with zinc acetate solution will eliminate some of these interferences. (Use approximately 15 drops of 2 N zinc acetate per 500 mL of sample if not already preserved with it.)

4.3. Samples must be taken with a minimum of aeration. Sulfide may be volatilized by aeration and any oxygen inadvertently added to the sample may convert the sulfide to an immeasurable form.

4.4. Samples that contain strong oxidizers or reducers will interfere with this method.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Iodine	Poison Corrosive Oxidizer	0.1 ppm-Ceiling	Vapors severely irritate and can burn the mucous membranes and respiratory tract. Liquid contact may cause blistering burns, irritation, and pain. Vapors may be severely irritating to the skin. Vapors are severely irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Sodium Hydroxide	Corrosive	2 mg/ m^3 - Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/ m^3 - TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose, throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Sodium Sulfide	Corrosive	10 ppm-TWA 15 ppm-STEL	Will form Hydrogen Sulfide (HS) gas if combined with strong acids. Inhalation of HS gas may be fatal. Symptoms include painful conjunctivitis, headache, nausea, dizziness, coughing and, in extreme cases, pulmonary edema and possible death. Irritant. Contact with skin can produce serious caustic burns with painful inflammation and possible destruction of tissue. Inflammation, tearing and pain may be expected. Severe contact can cause destruction of tissue.
1 – Always add acid to water to prevent violent reactions.			
2- Exposure limit refers to the OSHA regulatory exposure limit.			

5.3. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4. Sodium Sulfide will form Hydrogen Sulfide (HS) gas if combined with water moisture or strong acids. Inhalation of HS gas may be fatal.

5.6. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.7. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.8. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.9. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.10. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.11. Sulfide titration must be performed in a fume hood.

6. EQUIPMENT AND SUPPLIES

6.1. Volumetric pipettes: various

6.2. Burette: 10 mL and 25 mL Class A

6.3. Erlenmeyer flasks: 500 mL

6.4. Graduated cylinder: 50 mL and 200 mL

6.5. Top loading balance: capable of accurately weighing ± 0.01 g

6.6. Volumetric flasks: various

6.7. Distillation apparatus containing: Boiling tubes, inlet adapters, dropping/addition funnels, gas inlets, impinged bubblers, bubbler/scrubber vessels, Westclips®, gas line "T" connectors, Easy Midi Distillation heating block

6.8. Ottawa sand

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. 6N Hydrochloric Acid (1:1 HCl): Add 250 mL concentrated hydrochloric acid (HCl) to 250 mL of reagent water.

7.1.2. Starch Indicator: Add 10 mL of reagent water to 5 g starch (potato) and mix. Add starch mixture to 500 mL of boiling reagent water. Mix, cool, and store in a well-labeled squirt bottle. Alternately, use purchased starch solution.

7.1.3. 0.025 N Sodium Thiosulfate (stored in desiccator): Add 0.4 g NaOH and 6.205 g of sodium thiosulfate ($Na_2S_2O_3 \bullet 5H_2O$) to 500 mL of reagent water in a 1 liter volumetric. Dilute to volume with reagent water. Store in a dark container. This reagent is also available commercially.

7.1.3.1. Standardization of 0.025 N Sodium Thiosulfate Solution: *To make 0.025N Biodate Solution, dissolve 0.462 g $KH(IO_3)_2$ in 500 mL with reagent water. Weigh 2 g KI in a 500 mL Erlenmeyer flask. Add 100 to 150 mL reagent water, 5 drops H_2SO_4 and 20 mL Biodate solution using a volumetric pipette. Dilute to 200 mL with reagent water. Titrate with Sodium Thiosulfate. When a pale straw yellow color is reached, add 1-2 mL starch. Continue titrating from a blue to a clear end point.

Note: Biodate Solution may be purchased.

Calculation:

$$Na_xS_2O_3\ Normality = \frac{(a)\ (b)}{c}$$

Where:
a = mLs Biodate (20 mL)
b = Normality Biodate (0.025N)
c = mLs of $Na_2S_2O_3$ used to titrate Repeat two more times

7.1.4. 0.0282 N Iodine Solution: Add 20 g KI (potassium iodide) and 3.2 g iodine to a 1 liter volumetric flask. Add 500 - 700 mL of reagent water and dissolve. Dilute to volume with reagent water. Store in a dark container. This reagent is also available commercially.

7.1.4.1. Standardization 0.0282 N Iodine Solution: Perform three method blanks daily. Refer to method blank section in SOP.

Calculation:

$$\textbf{Normality Iodine} = \frac{(Normality\ Na_2S_2O_3)(mL\ of\ titrant\ Na_2S_2O_3)}{20\ mL\ Iodine}$$

7.1.5. Zinc Acetate Solution (approximately 0.5M): Dissolve 55g of zinc acetate in 400 mL reagent water. Add 0.5 mL HCl and dilute to 500 mL with reagent water.

7.1.6. Sulfuric acid – Concentrated.

7.1.7. Nitrogen – High Purity Grade

7.2. Standards

7.2.1. Laboratory Control Sample

7.2.1.1. 2000 ppm Sulfide: Add 3.5 g of sodium sulfide to 100 mL of reagent water in a 250mL volumetric flask. Dilute to volume with reagent water. The sulfide standard must be verified each working day. If the resulting value is <75% of the original standard, a new solution must be prepared.

7.2.2. Matrix Spike Standard

7.2.2.1. Prepare a midrange matrix spike standard as described in 7.2.1 for use as a MS/MSD.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Waters are preserved to a pH $>$ 9 with NaOH and zinc acetate. Non-water samples are unpreserved. All matrices are stored at 4°C $\pm$ 2°C in plastic or glass containers.

8.2. The holding time is seven days from sampling to analysis for aqueous and solid samples. Samples must be analyzed immediately if arrived in the laboratory without proper chemical preservation.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, and MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank (MB)

9.2.1. One MB must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The MB should not contain any analyte of interest at or above the reporting limit.

9.2.2 For Methods 376.1 and 4500-S2-F, a MB consisting of 200 mL reagent water must be analyzed with each analytical batch of samples.

9.2.3 For Method 9030B/9034, a MB consisting of 50 mL reagent water, or 5g Ottawa sand and 50 mL reagent water for solids and wastes, must be analyzed with each analytical batch of samples.

9.2.4 Corrective Action for MBs

9.2.4.1. If the analyte level in the MB exceeds the reporting limit for the analytes of interest in the sample, all associated samples are re-prepared and re-analyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.4.2. If there is no analyte greater than the RL in the samples associated with an unacceptable MB, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One aqueous LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. For Methods 376.1 and 4500-S2-F, a mid-range LCS is prepared by adding 2.5 mL of the 2000 ppm sulfide standard to 200 mL reagent water.

9.3.3. For Method 9030B/9034, a mid range LCS is prepared by adding 0.25 mL of 2000 ppm sulfide standard to 50mL reagent water or 5g Ottawa sand and 50 mL reagent water for solids or wastes.

9.3.4. LCS standard must be analyzed with each analytical batch of samples.

9.3.5. Corrective Action for LCS

9.3.5.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.5.2. The only exception is that if the LCS recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

9.3.5.3. Corrective action will be repreparation and re-analysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. For Methods 376.1 and 4500-S2-F, an MS/MSD consisting of 200 mL sample and 2.5 mL 2000 ppm sulfide standard must be analyzed with every 20 samples.

9.4.3. For Method 9030B/9034, an MS/MSD consisting of 50 mL sample for waters, 5g sample and 50 mL reagent water for solids and wastes, and 0.25 mL 2000 ppm sulfide standard must be analyzed with every 20 samples.

9.4.4. Corrective Action for MS/MSDs

9.4.4.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and re-analysis of the batch.

9.4.4.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported and flagged with a "4" in LIMS.

9.5. Control Limits

9.5.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.5.2. Laboratory control limits are internally generated and updated periodically unless method specified. The latest version is easily accessible via LIMs.

9.6. Method Detection Limits (MDLs) and MDL Checks

9.6.1. MDLs and MDL Checks are established by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.6.2. MDLs are easily accessible via LIMs.

9.7. Nonconformance and Corrective Action

9.7.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action.

10. CALIBRATION AND STANDARDIZATION

10.1. Not applicable

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. Aqueous Sample Method 9030B

11.3.1.1. For an efficient distillation, prepare the sample using one of the procedures in this section. Then proceed with the distillation step.

11.3.1.2. If the sample is aqueous, shake the sample container to suspend any solids, then quickly decant the appropriate volume (50 mL) of the sample to a graduated cylinder. Transfer the contents of the graduated cylinder into the boiling tube.

11.3.1.3. If the sample is aqueous, but contains a large proportion of solids, the sample may be roughly separated by phase and the amount of each phase measured and weighed to the nearest milligram into the distillation flask in proportion to their abundance in the sample. Reagent water may be added up to a total volume of 50 mL.

11.3.2. Solids and Waste Samples, Method 9030B

11.3.2.1. Weigh out 5g $\pm$ 0.1 g of homogenized sample and put into the reaction flask.

11.3.2.2. Samples that are not water miscible (oils, various solvents) cannot be analyzed using this method.

11.3.3. Method 9030B Distillation

11.3.3.1. Acid Soluble Sulfides

11.3.3.1.1. Place the boiling tube containing the sample in the heater block, and assemble the acid-soluble distillation apparatus as shown in Figure 1.

11.3.3.1.2 Place 2.0 mL of 0.5M zinc acetate solution and 25 mL reagent water in each of two bubbler/scrubber vessels. (The method also requires the addition of formaldehyde, but this has been found to cause interferences; therefore, it is not used.) Place an impinged bubbler (front) in the first vessel, a fritted bubbler (back) in the second vessel, and seal them with size 24/40 WestClips®. The sealed vessels and impingers function as the gas scrubbers. Connect the first scrubber to the inlet adapter, and place the second bubbler vessel in the bubbler vessel rack. Connect the two impingers in series using Tygon® tubing.

11.3.3.1.3 Close stopcock of addition/dropping funnel. Place 10 mL concentrated sulfuric acid in addition/dropping funnel.

11.3.3.1.4 Connect a high-purity (GC grade) nitrogen gas source to the main inlet of the gas manifold of the aluminum heater block as specified in the Heater Block Operation manual. Use a two-stage gas tank regulator, and set the pressure into the gas manifold to 20 psi.

11.3.3.1.5 Connect a black gas line from each gas manifold valve to a "T" connector and a Tygon® gas line from manifold valve to a "T" connector and a Tygon® gas line from the "T" to each of the two gas inlets of the apparatus--one at the top of the addition/dropping funnel, and one at the inlet adapter as shown in Figure 1.

11.3.3.1.6 Purge assembled apparatus with high-purity nitrogen for ten minutes to remove atmospheric oxygen from the apparatus and contained solutions. During purge, adjust nitrogen flow such that approximately five bubbles per second exit the base of the inlet adapter. A proper flow rate is indicated by a vigorous foaming action at the fritted bubbler.

11.3.3.1.7 Following instructions in the Easy Dist Heat Block manual set the program to "P1". Open the stopcock of the dropping/addition funnels and allow all the sulfuric acid to drop into the boiling tube at a rate of approximately 1 mL/min. Continue to observe the boiling tube contents as acid is added to monitor for excessive foaming, swelling of solids, or formation of a non-homogeneous mixture that is not suspended by the bubbling action in the tube. It may be necessary to use a smaller sample aliquot under such

conditions. Once dropping/addition funnel is empty, close the stopcock to ensure sample is not lost into the funnel. It may also be necessary to add antifoam to known swelling solids and waters.

11.3.3.1.8 After the 90-minute heating period, the heat will automatically shut off. Remove the bubbler vessels. Turn off the nitrogen flow. Carefully combine the gas scrubber solutions. Do not shake or mix solutions to avoid loss of sulfide. Determine the concentration of acid-soluble sulfide in the zinc acetate gas scrubber solutions by using the titrimetric-iodine method (9034).

11.4. Sample Analysis

11.4.1. Sample Analysis Procedure

11.4.1.1. Methods 376.1 and 4500-S2-F

11.4.1.1.1. Place 200 mL of homogenized sample in a 500 mL Erlenmeyer flask. At this time, add any spiking solutions if necessary for the filtered water samples.

11.4.1.1.2. Add 20.0 mL 0.028 N Iodine solution and approximately 2 mL 1:1 HCl solution (watch for fumes). Check the pH prior to titration to make sure it is less than 2. If the pH is not <2, add additional acid.

11.4.1.1.3. Add 1 squirt (1-2 mL) of starch indicator and mix. Titrate from blue to clear with 0.025 N sodium thiosulfate titrant. Record the amount of titrant used in LIMS.

Note: Some matrices may be turbid or colored and the color change from blue to clear may not be easily seen. In this case, look for a shade change.

11.4.1.1.4. After adding 20 mL iodine, the color should be orange/red. If the color remains yellow, add additional 20 mL aliquots until the orange/red color persists (adjust the calculation accordingly). If the sample requires more than 60 mL of iodine, the sample must be re-prepped at a dilution.

11.4.1.2. Methods 9030B/9034 Titrations

11.4.1.2.2 Add 8 mL of 0.0282 N iodine solution to scrubber solution and 1-2 mL 1:1 HCl (check pH prior to titration to make sure it is less than 2).

11.4.1.2.1. After adding 8 mL iodine, the color should be orange/red. If the color remains yellow, add additional 8 mL aliquots until the orange/red color persists (adjust the calculation accordingly). If the sample requires more than 24 mL of iodine, the sample must be re-prepped at a dilution.

11.4.1.2.2. Add 1 squirt of starch indicator and mix. Titrate from blue to clear with .025 N sodium thiosulfate titrant.

11.4.1.2.3. Using a 10 mL Class A burette, titrate scrubber solution in 150 mL disposable beaker.

11.5. Analytical Documentation

11.5.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.5.2. Record all standards and reagents in the LIMS Reagents module. All standards and reagents are assigned a unique number for identification.

11.5.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4. Record all sample results and associated QC directly into LIMS during analysis. Level I and Level II review is performed in LIMS.

11.6. Glassware Washing and Cleaning

11.6.1. Boiling tubes and bubbler/scrubber vessels must be cleaned with hot soapy water between each setup.

11.6.2. Impingers and inlet adapters must be cleaned with hot soapy water, and may require additional 1:1 HCl rinse if sulfide contamination is suspected. If 1:1 HCl rinse is required, many rinses with reagent water must follow to ensure all 1:1 HCl is completely rinsed away. Alternately, rinsing with 1N NaOH solution is acceptable.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Calculations

$$\textit{Sulfide, mg / L or mg / kg} = \frac{\textit{[(A x B) - (C x D)] x 16,000}}{\textit{mL or g of sample used}}$$

Where:
A = mL of iodine solution
B = Normality of iodine solution
C = mL of sodium thiosulfate titrant
D = Normality of sodium thiosulfate titrant

12.1.1.

$$LCS\,\%\ \text{Recovery} = \frac{\text{mg/L (from 12.1.1)}}{20\,(\text{true})} \text{ x } 100$$

Note: The true value of the standard is determined daily.

12.1.2.

$$\text{MS/MSD \% Recovery} = \frac{\text{A - B}}{20\,(\text{waters}) \text{ or } 1000\,(\text{solids})} \text{ x } 100$$

Where:
A = (20 - mL titrant for MS/MSD) x 400
B = Concentration from 12.1.1 x mL or g of sample used

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks followed by annual refresher training.

15.3. Waste Streams Produced by the Method

15.3.1. The following waste streams are produced when this method is carried out.

15.3.1.1. Acidic waste generated by sample titration. Waste material is disposed of in the 55-gallon waste stream called "Acid Waste" located in the lab.

16. REFERENCES

16.1. References

16.1.1. SW846, Test Methods of Evaluating Solid Waste, Third Edition, Sulfide, Method 9030B.

16.1.2. EPA 600, Methods for Chemical Analysis of Waters and Wastes, Sulfide (Titrimetric, Iodine), Method 376.1

16.1.3. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.4. Corporate Quality Management Plan (CQMP), current version

16.1.5. SW846, Test Methods of Evaluating Solid Waste, Third Edition, Titrimetric Procedure for Acid-soluble and Acid-insoluble Sulfides, Method 9034

16.1.6. Standard Methods, Sulfide, Iodometric Method 4500-S2-F, 2000.

16.1.7. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.8. Revision History

Historical File:		Revision 0: 02/14/96		Revision 6: 03/05/09
		Revision 1: 11/20/00		Revision 7: 11/16/10
		Revision 2: 09/17/02		
		Revision 3: 06/17/03		
		Revision 4: 09/24/04		
		Revision 5: 10/22/07		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5. Standards and Reagents, NC-QA-017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting Limits

17.1.1. The lower reporting limit for Methods 376.1 and 4500-S2-E is 1 mg/L for water. The acid-soluble sulfide reporting limit is 3 mg/L for water and 30 mg/kg for solid.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

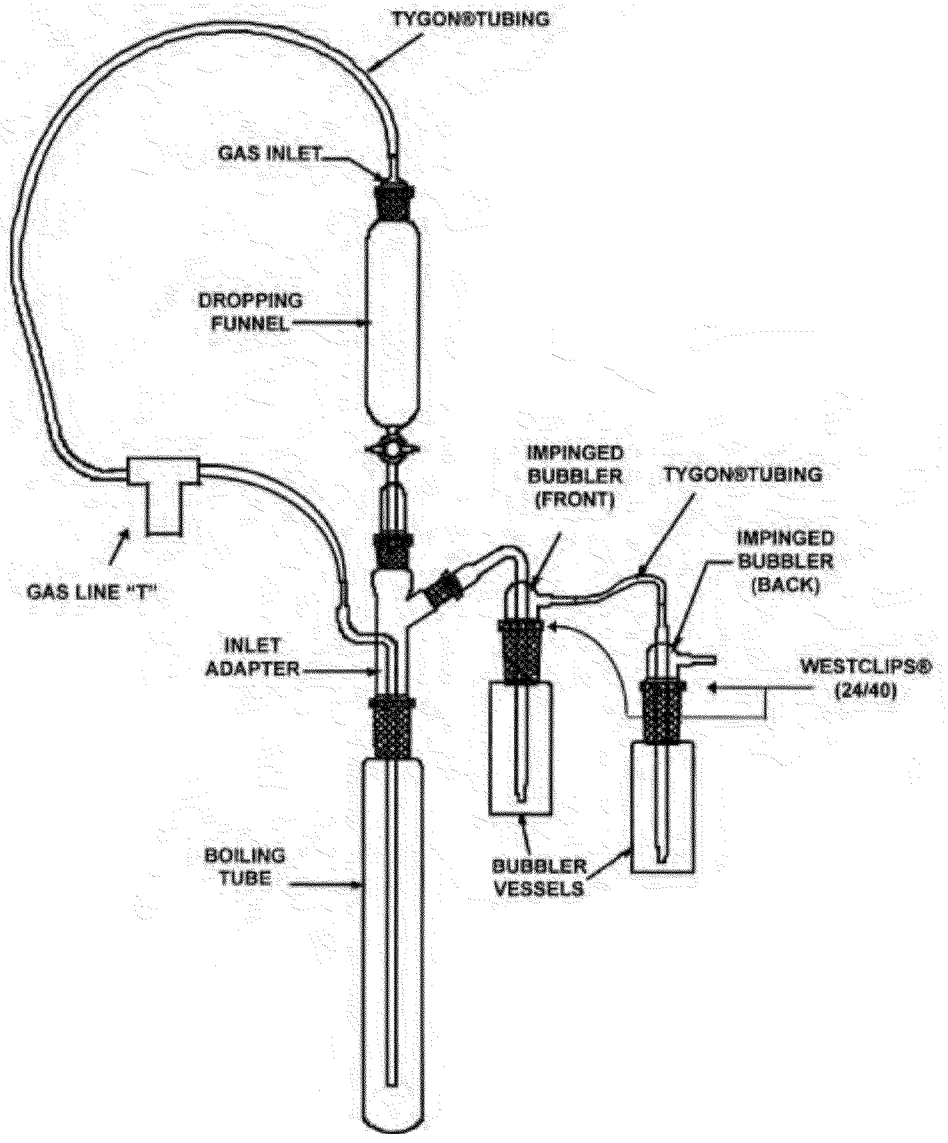
17.2. Method Deviations

17.2.1. The laboratory does not add formaldehyde due to interferences with the analysis.

17.2.2. The laboratory does not titrate the sample in the original container as specified in Method 376.1.

17.2.3. Methods 376.1, 9034, and SM4500 S2-F call for pipetting the sample below the surface of the iodine solution. The laboratory does not pipette the sample below the surface of the iodine solution.

FIGURE 1
ACID-SOLUBLE SULFIDE DISTILLATION APPARATUS



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica North Canton

SOP No. NC-WC-036, Rev. 3.4
Effective Date: 11/18/10

Title: TOTAL HARDNESS (mg/L $CaCO_3$), TITRIMETRIC

[Method: EPA Method 130.2 and Standard Method 2340C]

Approvals (Signature/Date):

[redacted]	11/08/10	[redacted]	11/02/10
Technology Specialist	Date	Health & Safety Coordinator	Date
[redacted]	11/08/10	[redacted]	11/02/10
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP NC-WC-036, Rev 3.3, dated 04/28/10

Facility Distribution No. _____ **Distributed To: _____**

TABLE OF CONTENTS

1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Hardness in waters and wastewaters. It is based on EPA Method 130.2 and Standard Method 2340C. The approximate working range is 2 to 400 mg/L.

1.2. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. Calcium and magnesium ions in the sample are sequestered upon the addition of magnesium ethylenediamine tetraacetate (MgEDTA). The end point of the reaction is detected using Eriochrome Black T indicator, which has a red color in the presence of calcium and magnesium and a blue color when the cations are sequestered.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Ammonium Hydroxide	Corrosive Poison	50 ppm-TWA	Vapors and mists cause irritation to the respiratory tract. Causes irritation and burns to the skin and eyes.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.7. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.8. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Stir plate and stir bars

6.2. Erlenmeyer flasks: various

6.3. Graduated cylinders: various

6.4. Autopipettor and disposable tips

6.5. Class A Microburet: 10 mL

6.6. Volumetric flasks: various

6.7. Plastic bottles: various

6.8. Analytical balance: Capable of accurately weighing ± 0.0001 g

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Ammonium Chloride (NH_4Cl): reagent grade

7.1.2. Ammonium Hydroxide (NH_4OH): reagent grade

7.1.3. Magnesium EDTA: reagent grade

7.1.4. EDTA Buffer Solution: Dissolve 16.9 g of NH_4Cl in 143 mL of NH_4OH. Add 1.25 g of magnesium salt of EDTA and dilute to 250 mL with reagent water. Mix well and store in a plastic bottle. This solution is stable for one month. Alternatively, use purchased hardness buffer.

7.1.5. Buffer for Water Hardness: Ammonium Chloride-Hydroxide Buffer with Magnesium EDTA.

7.1.6. Eriochrome Black T Indicator: reagent grade

7.1.7. Sodium Chloride (NaCl): reagent grade

7.1.8. Indicator: Mix together 0.5 g of Eriochrome Black T and 100 g of NaCl. Alternatively, use purchased indicator (such as Calamagite).

7.1.9. 1 N Ammonium Hydroxide: Dilute 7 mL of NH_4OH to 100 mL with reagent water and mix well.

7.1.10. Disodium EDTA Dihydrate: Primary standard grade

7.1.11. 0.02 N EDTA Titrant: Purchased. Alternatively, prepare titrant by dissolving 3.723 g of disodium EDTA dihydrate in about 400 mL of reagent water in a 1000 mL volumetric flash. Dilute to 1 liter with reagent water and mix well. This solution is stable for six months. Calibrate weekly against standard calcium solution by titration. Store in polyethylene bottle.

7.1.11.1. Standardization Titration Procedure: Place 5.0 mL of 1000 ppm standard calcium solution in a vessel containing about 50 mL of reagent water. Add 1 mL of buffer solution. Add about 0.5 - 1 g of dry indicator. Titrate slowly with continuous stirring until the last reddish tinge disappears, adding the last few drops at 3 - 5 second intervals. At the end point, the color is blue. Repeat the standardization procedure two more times and average the values.

$$\textbf{N of EDTA} = \frac{\textbf{0.1}}{\textbf{mL of EDTA}}$$

7.2. Standards

7.2.1. Calcium Carbonate Standard Solution, 1000 mg/L: Purchased standard (Hach or other supplier). Alternately, a standard may be made: Place 1.0 g of anhydrous calcium carbonate (primary standard grade) in a 500 mL Erlenmeyer flask. Slowly add 6 N HC1 until all the $CaCO_3$ has dissolved. Add 200 mL of reagent water and boil for five minutes. Cool the solution and add several drops of methyl red indicator. Adjust the solution to an intermediate orange color with 3 N NH_4 OH or 6 N HCl as required. Quantitatively transfer the solution to a 1 liter volumetric flask and dilute to volume with reagent water. This solution is stable for six months.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are preserved to a pH < 2 with concentrated nitric acid.

8.2. Samples are stored in plastic or glass containers at 4°C ± 2°C.

8.3. The holding time is six months from sampling to the completion of analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD and Sample Duplicates) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.2. A reagent water blank consisting of 50 mL of reagent water and all other reagents added to samples must be analyzed with each analytical batch of samples.

9.2.3. Corrective Action for Blanks

9.2.3.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and re-analyzed. If this is not possible due to limited sample quantity or holding time considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.3.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One aqueous LCS from an independent source must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. A midrange LCS must be analyzed with each analytical batch of samples. The LCS is made of 45 mL reagent water and 5.0 mL 1000 ppm $CaCO_3$ standard. Alternatively, use 25mL water and 25mL purchased hardness standard.

9.3.3. Corrective Action for LCS

9.3.3.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.3.2. The only exception is that if the LCS recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

9.3.3.3. Corrective action will be repreparation and re-analysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. An MS/MSD must be analyzed per analytical batch. The MS/MSD is composed of 20mL sample, 25mL reagent water, and 5mL 1000ppm $CaCO_3$.

9.4.3. Corrective Action for MS/MSDs

9.4.3.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and re-analysis of the batch.

9.4.3.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported as "amount" MSB. The Exception Code is changed to NC. The following two footnotes

will appear on the report page "NC The recovery and/or RPD were not calculated." "MSB The recovery and RPD were not calculated because the sample amount was greater than four times the spike amount."

9.4.3.3. In an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the laboratory matrix spike RPD limits.

9.4.3.4. If client program requirements specify to confirm matrix interference's, repreparation and re-analysis of the MS/MSD may be necessary.

9.5 Sample Duplicate

9.5.1 Sample duplicates are performed at a frequency of 10% and must meet laboratory-specific limits for precision.

9.6 Control Limits

9.6.1 Control limits are established by the laboratory as described in NC-QA-018.

9.6.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMs (QC Browser program).

9.7 Method Detection Limits (MDLs) and MDL Checks

9.7.1 MDLs and MDL checks are established by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.7.2 MDLs are easily accessible via LIMs (QC Browser program).

9.8 Nonconformance and Corrective Action

9.8.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager.

10 CALIBRATION AND STANDARDIZATION

10.7 Not applicable

11 PROCEDURE

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. The Nonconformance Memo shall be filed in the project file.

11.2 Any unauthorized deviations from this procedure must also be documents as a nonconformance with a cause and corrective action described.

11.3 Sample Preparation

Not applicable

11.4 Sample Analysis Procedure

11.4.1 All reagents and samples must be at ambient temperature prior to analysis.

11.4.2 Rinse the burette with reagent water once and with EDTA titrant once. Fill with EDTA titrant and be sure all air bubbles are removed.

11.4.3 Place 25 mL of sample and 25 mL of reagent water in an Erlenmeyer flask. Add approximately 1 to 2 mL of 1N NH_4OH, approximately 1 to 2 mL of EDTA buffer, (or add 3-4mL of the purchased buffer) and approximately 0.5 to 1 g of Indicator. Titration should be completed within 5 minutes of the addition of the buffer.

11.4.4 While stirring, slowly titrate with EDTA titrant until the last reddish tint disappears. The end point is blue. Record the volume of titrant used and its true normality on the analytical logsheet.

NOTE: Samples requiring more than 10 mL of titrant must be diluted. An aliquot is diluted to 25 mL with reagent water and treated as a sample.

11.5 Analytical Documentation

11.5.1 Record all analytical information in the analytical logbook/logsheet which may be in an electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2 All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3 Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4 Sample results and associated QC are entered into LIMs after final technical review.

12 DATA ANALYSIS AND CALCULATIONS

12.1 Hardness, mg/L $CaCO_3$ = $\frac{\mathbf{(A - B) \times N \times 50{,}000}}{\mathbf{C}} \times \mathbf{D}$

Where:
A = Volume of EDTA titrant used for sample, mL
B = Volume of EDTA titrant used for blank, mL
C = Volume of sample used, mL (not total volume after addition of DI water)

D = Dilution Factor = $\frac{\textit{Final Dilution Volume, mL}}{\textit{Initial Sample Volume Used For Dilution, mL}}$

N = Normality of EDTA titrant
50,000 = 50 (Equivalent weight of Calcium) x 1000 mg/g

12.1.1 LCS Recovery

$$\textbf{LCS \% Recovery} = \frac{\textbf{mL titrant x N x 50,000}}{\textbf{(25) LCS TV}} \times \textbf{100}$$

Where:
N = Normality of EDTA titrant
LCS TV = value of purchased hardness standard

12.1.2 MS/MSD Calculation

$$\textbf{MS / MSD \% Recovery} = \frac{\textbf{(A – B)}}{\textbf{200}} \times \textbf{100}$$

Where:
A = MS/MSD concentration (from 12.1)
B = Sample concentration (from 12.1)

13 METHOD PERFORMANCE

13.1 Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2 Training Qualifications

13.2.1 The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2 Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14 POLLUTION PREVENTION

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15 WASTE MANAGEMENT

15.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2 Waste Streams Produced by the Method

15.2.1 The following waste streams are produced when this method is carried out.

15.2.1.1 **Aqueous waste generated by the analysis.** Aqueous waste can be poured down the drain if the pH is between 5 and 10. Any sample waste generated that is not in this pH range must be collected and disposed of in the acid waste drum labeled as "Acid Waste".

15.3 Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica

North Canton. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks followed by annual refresher training.

16 REFERENCES

16.1 References

16.1.1 EPA 600, Methods for Chemical Analysis of Water and Wastes, Hardness, Total (mg/L as $CaCO_3$, Titrimetric, EDTA), Method 130.2

16.1.2 Standard Method for EDTA Titrimetric Method, 18th Edition, Method 2340C

16.1.3 TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.4 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.5 Corporate Quality Management Plan (CQMP), current version

16.1.6 Revision History

Historical File:		Revision2: 12/23/98		
		Revision3: 04/19/99		
		Revision 3.1: 11/06/04		
		Revision 3.2: 03/21/08		
		Revision 3.3: 04/28/10		

16.2Associated SOPs and Policies, current version

16.2.1 Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.2 QA Policy, QA-003

16.2.3 Glassware Washing, NC-QA-014

16.2.4 Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5 Supplemental Practices for DoD Project Work, NC-QA-016

17 MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1 Reporting Limits

17.1.1 The lower reporting limit (RL) for undiluted samples is 5 mg/L $CaCO_3$.

17.1.2 If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2 Deviation from EPA Method 130.2 and Standard Method 2340C:

17.2.1 Wastewater samples are not digested.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

North Canton

SOP No. NC-WC-034, Rev. 2
Effective Date: 03/21/13

Title: FLASHPOINT CLOSED CUP

[Method: SW846 Method 1010, 1010A, and ASTM D93-80]

Approvals (Signature/Date):

Technology Specialist	3/21/2013 Date	Health & Safety Coordinator	3/21/2013 Date
Quality Assurance Manager	3/21/2013 Date	Laboratory Director	3/21/2013 Date

This SOP was previously identified as SOP No. NC-WC-0034, Rev 1.3, dated 04/09/12

Facility Distribution No. _____ **Distributed To:_____**

TABLE OF CONTENTS

1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Flashpoint by Pensky-Martens closed cup tester in a variety of wastes, liquids, and solids. It is based on ASTM D93-80, SW846 Method 1010, and 1010A. Although the approximate working range is 20-200 °F, the test is generally considered complete when the temperature reaches 180°F without a measurable Flashpoint.

1.2. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. The sample is heated at a slow constant rate with continual stirring if it is a liquid or water. A small test element is directed into the sample cup at regular intervals. The Flashpoint is the lowest temperature at which the test element causes the vapor above the sample to ignite.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Not Applicable

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
p-Xylene	Flammable Irritant	100 ppm-TWA	Inhalation of vapors may be irritating to the nose and throat. Inhalation of high concentrations may result in nausea, vomiting, headache, ringing in the ears, and severe breathing difficulties, which may be delayed in onset. High vapor concentrations are anesthetic and central nervous system depressants. Skin contact results in loss of natural oils and often results in a characteristic dermatitis. May be absorbed through the skin. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves MUST be worn when doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.7. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.8. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to a Laboratory Supervisor and the EH&S Coordinator.

5.9. In the event a sample ignites in the test apparatus do not attempt to remove the sample. Turn off the apparatus and flame. The flame should go out when the cup is closed. If this does not happen the flame may be extinguished by covering the sample with a non-flammable material. After the apparatus has cooled the sample may be removed.

5.10. **When testing a sample, the analyst shall remain within eyesight of the flash point tester and will manually shut down the tester if it fails to automatically shut down following ignition.**

6. EQUIPMENT AND SUPPLIES

6.1. Ignitor and detector

6.2. Pensky-Martens closed cup tester with stirrer, stirring motor, internal barometer, and thermocouple

6.3. Flash point sample cup with lid

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. p-Xylene

8. SAMPLE COLLECTION, PRESERVATIONAND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored in glass containers at 4°C ± 2°C. Samples are not to be stored in plastic containers since volatile materials may diffuse through the walls of the enclosure.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS and Method Blank) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Laboratory Control Sample (LCS)

9.2.1 One LCS must be processed with each analytical batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process and the accuracy of the Flashpoint Apparatus. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.2.2 One LCS consisting of p-Xylene will be analyzed at the beginning of every batch. P-Xylene has a flashpoint of 81 °F. If the flashpoint of the p-Xylene varies by more than $\pm$ 2 °F, corrective action is required before samples can be analyzed.

NOTE: Dependent upon ambient temperature, p-Xylene may have to be cooled to just above the freezing point in order to achieve accurate results.

9.2.3 Corrective Action for LCS

9.2.3.1 If the p-Xylene flashpoint temperature is outside of established control limits, the system is out of control and corrective action must occur.

9.2.3.2 Corrective action consists of troubleshooting the apparatus for errors and cleanliness, followed by re-analysis of the p-Xylene LCS.

9.3. Duplicates

9.3.1. A sample duplicate (DU) is a second aliquot of an environmental sample, taken from the same sample container when possible, that is processed with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. The sample and DU results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample/DU precision results are not necessarily representative of the precision for other samples in the batch.

9.3.2. Sample duplicates are performed at a minimum frequency of 10% per matrix () and must meet laboratory-specific limits for precision. Soil and Waste matrices may be combined for batching purposes.

9.4. Control Limits

9.4.1. For this test method, the RPD value is used to assess the sample duplicate.

9.5. Nonconformance and Corrective Action

9.5.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration of tester

10.1.1. Determine the flash point of p-Xylene following the procedures outlined in Section 11.3.

10.1.2. The tester is operating properly when a value of 81± 2°F is obtained (See section 9.2).

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Non-Conformance Memo. The Non-Conformance Memo shall be filed in the project file in LIMS.

11.2. Sample Preparation Procedure

11.2.1. Not Applicable

11.3. Sample Analysis Procedure

11.3.1. Operate the Pensky-Martens tester according to the manufacturer's specifications, in a well-ventilated area away from flammable materials and significant air movement. Operating manuals are located in the laboratory near the tester. Performing this analysis under a hood is the best approach - ensure that the air intake and hood lights are turned off. Inspect the igniter coil closely to ensure the filaments are not touching. Proper placement of the igniter coil in the flashpoint apparatus is crucial to achieving accurate results. Fill the sample cup to the designated line with sample, and assemble the tester as directed.

11.3.1.1. Solid samples require a modified cover for the sample cup. The solid material is placed in the cup until even with the designated line. The sample is not stirred.

11.3.2. A flash check (LCS) must be analyzed with each analytical batch of samples. The compound p-Xylene is used to provide the analyst a reference true flash. The flash temperature is recorded in LIMS. - The true flash temperature for p-Xylene is 81°F. The acceptable range for p-Xylene flashpoint is 79 to 83°F. If the p-Xylene flashpoint is outside of this range, troubleshoot the system and rerun the p-Xylene. Sample analysis will not proceed until a passing p-Xylene flashpoint is achieved.

11.3.3. Pour the sample into the cup, assemble the apparatus, and record the initial temperature. Start the flashpoint apparatus.

11.3.3.1. If sample flashes, record the temperature at which the flash occurred in LIMS. Repeat sample for confirmation using the observed flashpoint as the estimated flashpoint.

NOTE: Confirmation analysis is run using a program that is specifically built for known flashpoints. Reference the apparatus user manual to chose the appropriate program.

11.3.3.2. If the temperature reaches 180°F and no flash occurs (see manual), turn off flashpoint and record > 180°F as the flashpoint in LIMS.

NOTE: Some samples require an upper limit flashpoint detection of 200 °F. This will be noted in the method comments. Samples with these limits that do not flash should be reported as > 200 °F

11.3.4. If solvents are used to clean sample cups, be sure to clean thoroughly with reagent water to prevent contamination.

NOTE: Some samples may burn, but not flash. If possible, record the initial temperature at which it burns in the comments section of the worksheet. Report these samples as "DNF" with an NCM.

Some samples have a flashpoint below room temperature. If this is known, the sample should be chilled to just above freezing and then analyzed promptly to confirm a true flashpoint.

11.4. Analytical Documentation

11.4.1. Record all analytical information in LIMS including the analytical data from standards and any corrective actions or modifications to the method.

11.4.2. All standards are logged into the LIMS standards and reagents module.

11.4.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.4.4. Sample results and associated QC are entered into the LIMs where a first and second level review will be done.

12. DATAANALYSISAND CALCULATIONS

12.1. If the sample did not flash, report > 180°F or > 200 °F as requested.

12.2. If the sample flashes, record and report the observed flashpoint.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications

13.2.1. The Group/Team Leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. Refer to the Laboratory Sample and Waste Disposal plan.

15.2. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica North Canton. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

15.3. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.4. Waste Streams Produced by the method

15.4.1. The following waste streams are produced when this method is carried out.

15.4.1.1. **Solid samples.** Solids are put into the red can for the debris waste stream

15.4.1.2. **Liquid samples and waste solvents.** Flammable wastes including the Xylene standard are disposed of in the solvent waste stream located in the red can in the hood. Adding water to the solvent waste stream should be avoided.

16. REFERENCES

16.1. References

16.1.1. Annual Book of ASTM Standards, ASTM Method D93-80, Flashpoint by Pensky-Martens Closed Tester

16.1.2. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Pensky-Martens Closed Cup Method for Determining Ignitability, Method 1010, Revision 0, September 1986.

16.1.3. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Pensky-Martens Closed Cup Method for Determining Ignitability, Method 1010A, Revision 1, November 2004.

16.1.4. Corporate Quality Management Plan (CQMP), current version

16.1.5. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.6. Revision History

Historical File:		Revision 0: 10/24/97		Revision 1.2: 09/18/09
		Revision 1.0: 09/25/03		Revision 1.2: 04/09/12
		Revision 1.1: 06/28/07		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Standards and Reagents, NC-QA-017

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-016

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Deviations from Method

17.1.1. Methods SW846 1010, 1010A, and ASTM D93-80 do not reference solid materials. Section 11.3.1.1 notes the procedure for solid samples.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Sacramento

SOP No. WS-ID-0005, Rev. 7.5
Effective Date: 04/19/2013
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Title: Analysis of Samples for Polychlorinated Dioxins and Furans by HRGC/HRMS

[Methods 8290, 8290A & TO-9A]

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1. SCOPE AND APPLICATION

1.1.1. This method provides procedures for the detection and quantitative measurement of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD), polychlorinated dibenzo-p-dioxins (tetra- through octachlorinated homologs; PCDDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologs; PCDFs) in a variety of environmental matrices at part-per-trillion (ppt) concentrations by SW 846 Method 8290 and 8290A. The analytical method calls for the use of high-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. An optional method for reporting the analytical results using a 2,3,7,8-TCDD toxicity equivalency factor (TEF) is also described. Table 1 lists the various sample types covered by this analytical protocol, the 2,3,7,8-TCDD-based method calibration limits and other pertinent information.

1.2. The sensitivity of this method is dependent upon the level of interferences within a given matrix.

1.3. This method is designed for use by analysts who are experienced with residue analysis and skilled in high-resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS).

1.4. Samples containing concentrations of specific congeners (PCDDs and PCDFs) that are greater than the calibration limit should be analyzed by a protocol designed for such concentrations, such as 8280A/B.

1.5. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 "DoD QSM and AFCEE QAPP Implementation" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. This procedure uses matrix-specific extraction, analyte-specific cleanup, and high-resolution capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS) techniques. Sample preparation is addressed in WS-IDP-0005.

2.2. One to two µL of the concentrated extract are injected into an HRGC/HRMS system capable of performing selected ion monitoring at resolving powers of at least 10,000 (10 percent valley definition).

2.3. The identification of ten of the 2,3,7,8-substituted congeners (Table 3), for which a ^{13}C-labeled standard is included as a spiked compound, is based on their elution at their exact retention time (-1 to +3 seconds from the respective isotope dilution analyte or internal standard signal) and simultaneous detection of the two most abundant ions in

the molecular ion region. All other identified PCDD/PCDF congeners are identified by their RRT's based on the daily CCV standard, and the simultaneous detection of the two most abundant ions in the molecular ion region. Confirmation is based on a comparison of the ratio of the integrated ion abundance of the molecular ion species to their theoretical abundance ratio.

2.4. Quantification of the individual congeners, total PCDDs and total PCDFs is achieved in conjunction with the establishment of a multipoint (five points) calibration curve for each homolog, during which each calibration solution is analyzed once.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

3.3. Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs): compounds (Figure 1) that contain from one to eight chlorine atoms. The seventeen 2,3,7,8-substituted PCDDs and PCDFs are shown in Table 3. The number of isomers at different chlorination levels is shown in Table 4.

3.4. Homologous series: Defined as a group of chlorinated dibenzodioxins or dibenzofurans having a specific number of chlorine atoms.

3.5. Isomer: Chemical compounds that contain the same number of atoms of the same elements, but differ in structural arrangement and properties. For example, 1,2,3,4-TCDD and 2,3,7,8-TCDD are different structural isomers.

3.6. Congener: Any isomer of any homologous series.

3.7. Isotope Dilution Analyte: An isotope dilution analyte is a ^{13}C-labeled analog of a congener chosen from the compounds listed in Table 3. Isotope dilution analytes are added to all samples including method blanks and quality control samples before extraction, and they are used to quantitate the concentration of the analytes. Nine isotope dilution analytes are used in this method. There is one for each of the dioxin and furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight. Additional isotope dilution analytes may be added to act as retention time references, but they are not used for quantitation.

3.8. Internal Standard: Two internal standards are used to determine the percent recoveries for the isotope dilution analytes. The ^{13}C-1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated isotope dilution analytes while ^{13}C-1,2,3,7,8,9-HxCDD is used to determine the recovery of the hexa-, hepta- and

octachlorinated isotope dilution analytes. ^{13}C-1,2,3,7,8,9-HxCDD also acts as a retention time reference for the unlabeled analog present in sample extracts. They are added to the final sample extract before HRGC/HRMS instrument analysis.

3.9. Estimated Detection Limit (EDL)/ Estimated Quantiation Limit (EQL): The sample specific estimated detection limit (EDL/EQL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background noise level.

3.10. Estimated Maximum Possible Concentration (EMPC): The calculated concentration of a signal having the same retention time as a PCDD/PCDF congener, but which does not meet the other qualitative identification criteria defined in the method.

4. INTERFERENCES

4.1. Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferents under the conditions of analysis by running laboratory method blanks. Analysts shall not use PVC gloves.

4.2. The use of high-purity reagents and solvents helps minimize interference problems. Purification of solvents by distillation in all-glass systems may be necessary.

4.3. Re-use of glassware is to be minimized to avoid the risk of contamination.

4.4. Interferents co-extracted from the sample will vary considerably from matrix to matrix. PCDDs and PCDFs are often associated with other interfering chlorinated substances such as polychlorinated biphenyls (PCBs), polychlorinated diphenyl ethers (PCDPEs), polychlorinated naphthalenes, and polychlorinated xanthenes that may be found at concentrations several orders of magnitude higher than the analytes of interest. Retention times of target analytes must be verified using reference standards. These values must correspond to the retention time windows established. While certain clean-up techniques are provided as part of this method, unique samples may require additional cleanup steps to achieve lower detection limits.

4.5. A high-resolution capillary column (60m DB-5) is used to resolve as many PCDD and PCDF isomers as possible. However, no single column is known to resolve all isomers. The DB-225 column is used for the quantitation of 2,3,7,8-TCDF when 2,3,7,8-TCDF on the DB-5 column is detected.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S

Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. The effluents of sample splitters for the gas chromatograph and roughing pumps on the HRGC/HRMS system should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohols.

5.1.2. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile or similar gloves must be used. Latex gloves may be used for methanol.

5.1.3. Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.4. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Iso-octane	Flammable Irritant	None established	Inhalation of vapors may cause nausea, headache, dizziness, loss of consciousness, irritation to upper respiratory tract, pain in throat and nose, coughing, wheezing, shortness of breath.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Tetradecane	Irritant	None established	Inhalation of vapors may cause difficulty breathing, headache, intoxication and central nervous system damage.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Preventive and routine maintenance is described in the 'Schedule of Routine Maintenance' in the QAM.

6.2. High-Resolution Gas Chromatograph/High-Resolution Mass Spectrometer/Data System (HRGC/HRMS/DS).

6.2.1. Capable of collecting, recording and storing MS data. The VG70 and Autospec Ultima systems utilize Opus version 3.6 software and the Autospec Premiere system utilizes MassLynx version 4.1 software.

6.2.2. The GC must be equipped for temperature programming. All required accessories must be available, such as syringes, gases, and capillary columns. The GC injection port must be designed for capillary columns. The use of splitless injection techniques is recommended. The use of a moving needle

injection port is also acceptable. When using the method described in this protocol, a 2-µL injection volume is used consistently (i.e., the injection volumes for all extracts, blanks, calibration solutions and the performance check samples are 2 µL). 1 µL injections are allowed; however, laboratories are encouraged to remain consistent throughout the analyses by using the same injection volume at all times on a given HRGC/HRMS/DS.

6.2.3. Gas Chromatograph/Mass Spectrometer (GC/MS) Interface - The GC/MS interface components should withstand 350° C. The interface must be designed so that the separation of 2,3,7,8-TCDD from the other TCDD isomers achieved in the gas chromatographic column is not appreciably degraded. Cold spots or active surfaces (adsorption sites) in the GC/MS interface can cause peak tailing and peak broadening. It is recommended that the GC column be fitted directly into the mass spectrometer ion source without being exposed to the ionizing electron beam. Graphite ferrules should be avoided in the injection port because they may adsorb the PCDDs and PCDFs. Vespel® or equivalent ferrules are recommended.

6.2.4. Mass Spectrometer - The static resolving power of the instrument must be maintained at a minimum of 10,000 (10 percent valley). The mass spectrometer must be operated in a selected ion monitoring (SIM) mode with a total cycle time (including the voltage reset time) of one second or less.

6.2.5. Data System - A dedicated data system is employed to control the rapid multiple ion monitoring process and to acquire the data. Quantification data (peak areas or peak heights) and SIM traces (displays of intensities of each ion signal being monitored including the lock-mass ion as a function of time) must be acquired during the analyses and stored. Quantifications may be reported based upon computer-generated peak areas or upon measured peak heights (chart recording). The data system must be capable of acquiring data for a minimum of 10 ions in a single scan. It is also recommended to have a data system capable of switching to different sets of ions (descriptors) at specified times during an HRGC/HRMS acquisition. The data system should be able to provide hard copies of individual ion chromatograms for selected gas chromatographic time intervals. It should also be able to acquire mass-spectral peak profiles and provide hard copies of peak profiles to demonstrate the required resolving power. The data system should also permit the measurement of noise on the base line.

6.3. GC Column

6.3.1. Due to poor separation of 2,3,7,8-TCDF from other TCDF isomers on the 60 m DB-5 column, a 30M DB-225 is used to quantitate 2,3,7,8-TCDF. This column is used when 2,3,7,8-TCDF is detected.

6.3.2. In order to have an isomer-specific determination for 2,3,7,8-TCDD and to allow the detection of OCDD/OCDF within a reasonable time interval in one HRGC/HRMS analysis, the 60-m DB-5 fused-silica capillary column is recommended. At the beginning of each 12-hour period during which samples are analyzed and after tuning, acceptable compound separation on the GC column must be demonstrated through the analysis of a column performance check solution. Operating conditions known to produce acceptable results with the recommended column are shown in Table 7.

7. REAGENTS AND STANDARDS

7.1. Solvents

7.1.1. High-purity, distilled-in-glass or highest available purity: methylene chloride, hexane, methanol, tetradecane, isooctane, toluene, and acetone.

7.2. All calibration, daily isotope dilution analyte, daily clean up internal standards, and daily spiking solutions are stable for one year from preparation. After 1 year, solutions may be re-verified. The re-verified solution may be used for an additional year, or until there is evidence of compound degradation or concentration. The re-verification must be performed using an unexpired, not previously re-verified solution from a second lot or second vendor.

7.2.1. Sealed ampules may be used until the manufacturer's expiration date is exceeded. If no expiration date is provided, then the expiration date will be 10 years from the date the ampule is opened. The solvent level should be monitored prior to each use to assure there has been no concentration of the standard over time.

7.3. Calibration Solutions

7.3.1. High-Resolution Concentration Calibration Solutions (Table 5) - Five tetradecane solutions containing unlabeled (totaling 17) and carbon-labeled (totaling 16) PCDDs and PCDFs at known concentrations are used to calibrate the instrument. The concentration ranges are homolog dependent, with the lowest values associated with the tetra chlorinated dioxins and furans (0.5 pg/μL) and the highest for the octachlorinated congeners (2000 pg/μL).

7.3.2. Individual isomers that make up the high-resolution concentration calibration solutions are obtained from commercial sources and prepared in the laboratory. These standards are traceable back to EPA-supplied standard solutions.

7.3.3. Store the calibration solutions in appropriate containers and at room temperature in the dark.

7.3.4. Standards for method 8290A require storage at ≤ 6°C.

7.4. GC Column Performance Check Solution

7.4.1. This solution contains the first and last eluting isomers for each homologous series from tetra- through hepta-chlorinated congeners. The solution also contains a series of other TCDD isomers for the purpose of documenting the chromatographic resolution. The ^{13}C-2,3,7,8-TCDD is also present. The laboratory is required to use tetradecane as the solvent and adjust the volume so that the final concentration does not exceed 100 pg/µL per congener. Table 8 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution for the DB-5 column.

7.4.2. For the DB-225 column, the column performance check solution contains a series of TCDF isomers in addition to the 2,3,7,8-TCDF. The solution is injected and evaluated at the start of each analytical sequence on the DB-225 column to ensure that 2,3,7,8-TCDF is resolved from its closest eluting isomers with a baseline-to-valley ratio of ≤ 25%. Table 8 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution on for the DB-225 column.

7.5. Field Surrogate Solution (air matrices)

7.5.1. This solution contains one ^{37}Cl labeled analog (for Method TO-9/TO-9A) or one ^{37}Cl and four ^{13}C labeled analogs (for Methods 23 and/or 0023A) at the nominal concentration indicated in Table 2. It is used to assess sample collection and recovery procedures.

7.6. Sample Fortification Solution (Isotope dilution analyte)

7.6.1. This isooctane (or toluene) solution contains the nine isotope dilution analytes at the nominal concentrations that are listed in Table 2. The solution contains at least one carbon-labeled standard for each homologous series, and it is used to measure the concentrations of the native substances. (Note that ^{13}C-OCDF is not present in the solution.)

7.7. Internal Standard Solution

7.7.1. This tetradecane solution contains two internal standards (^{13}C-1,2,3,4-TCDD and ^{13}C-1,2,3,7,8,9-HxCDD). An appropriate volume of this solution will be spiked into each sample extract before the final concentration step and HRGC/HRMS analysis.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. The sample collection, shipping, handling, and chain-of-custody procedures are not described in this document. Sample collection personnel will, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory. The analyst should make a judgment, based on the appearance of the sample, regarding the necessity for additional mixing. If the sample is clearly non-homogeneous, the entire contents should be transferred to a glass or stainless steel pan for mixing with a stainless steel spoon or spatula before removal of a sample portion for analysis.

8.2. Grab and composite samples must be collected in glass containers.

8.3. Ambient air samples are collected on a Quartz Fiber Filter followed by a glass sleeve containing a polyurethane foam plug.

8.4. Samples from stationary sources are collected on glass or quartz fiber filters and XAD-2 Resin. (See WS-ID-0009 for sample preparation procedures).

8.5. Conventional sampling practices must be followed. Do not rinse the bottle with sample before collection. Sampling equipment must be free of potential sources of contamination.

8.6. With the exception of the fish tissues, which must be stored at - 20°C, all samples should be stored at 4°C ± 2, extracted within 30 days and completely analyzed within 45 days of collection. The 30 day hold time is recommended. PCDDs and PCDFs have demonstrated stability for greater than one year.

8.7. All extracts must be stored capped, in the dark, at room temperature (approximately 21°C to 28°C). All extracts for method 8290A must be stored capped at $\leq 6^{o}$C.

9. QUALITY CONTROL

9.1. One method blank (MB) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory matrix (reagent water, Ottawa sand, sodium sulfate, PUF, XAD, filter, etc.) processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. The method blank contains a PUF plug, XAD, or filter prepared from the same batch as the field samples whenever possible for air samples.

Certain programs, such as DOD, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the lower calibration limit.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.1.1. The method blank must be spiked prior to extraction with the same amount of ^{13}C-labeled isotope dilution analytes as added to samples.

9.1.2. If method blank contamination is present, check solvents, reagents, fortification solutions, apparatus and glassware to locate and eliminate the source of contamination before any further samples are extracted and analyzed.

9.1.2.1. OCDD is a ubiquitous laboratory contaminant. A method blank and the associated samples are deemed acceptable if the OCDD concentration is <5x the specified reporting limit. Flag data appropriately. The analyst is expected to investigate and eliminate potential sources of systematic contamination.

9.1.2.2. If a target analyte is detected in the blank but the associated samples are ND (not detected), then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.2.3. If a target analyte is detected in the blank, but the concentration of the contaminant in the samples >10x the blank concentration, then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.3. If new batches of reagents or solvents contain interfering contaminants, purify or discard them.

9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, Ottawa sand, sodium sulfate, PUF, XAD, etc.) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See policy WS-PQA-003 for specific acceptance criteria. When associated with PUF samples, the LCS should contain a PUF plug prepared from the same batch as the field samples whenever possible.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.2.1. A LCS is deemed acceptable if control analytes are above upper control limits and the associated samples are ND, unless otherwise specified by the client. Note any actions in the narrative.

9.3. The assessment of matrix effects on method performance, as required by NELAP, is met in Method 8290 and 8290A, as in all isotope dilution techniques, with the use of isotopically labeled compounds. These isotopically labeled compounds are analogs of target analytes and are spiked into each sample. Therefore, matrix effects on method performance may be judged by the recovery of these analogs. Sample analysis acceptance is controlled by the performance of these analogs in each sample. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair are extracted at the client's request only. Method 8290A does not address analysis of MS/MSD. An exception to this rule is a batch containing South Carolina samples for Method 8290. These batches must have an MS/MSD prepared. However, South Carolina requires Method 8290A after December 31, 2008. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. When requested by the client, the MS/MSD pair shall be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, the LCS, the selected field sample, and the MS/MSD may be required after evaluation and review. Matrix Spike/ Matrix Spike Duplicates are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. An LCS/LCSD may be extracted to show precision of the extraction and analysis process.

9.3.1. Matrix Spike (MS): A sample, which is spiked with a known amount of the matrix spike fortification solution prior to the extraction step. The recoveries of the matrix spike compounds are determined; they are used to estimate the effect of the sample matrix upon the analytical methodology.

9.3.2. Matrix Spike Duplicate (MSD): A second portion of the same sample as used in the matrix spike analysis and which is treated like the matrix spike sample.

9.3.3. Locate the sample for the MS and MSD analyses (the sample may be labeled "double volume").

9.3.4. Add an appropriate volume of the matrix spike fortification solution, adjusting the fortification level as specified in Table 1, under IS Spiking Levels.

9.3.5. Analyze the MS and MSD samples as described in Section 11.

9.3.6. The results obtained from the MS and MSD samples (percent recovery and concentrations of 2,3,7,8-substituted PCDDs/PCDFs) should agree within 20 percent relative difference. Report all results and flag outliers.

9.3.7. Isotope dilution analyte recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any isotope dilution analyte is less than 10:1.

9.4. Duplicates

9.4.1. Upon client request, duplicates may be processed. Locate the sample specified for duplicate analysis, and prepare and analyze a second 10-g soil or sediment sample portion or 1-L water sample, or an appropriate amount of the type of matrix under consideration. Duplicate samples are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. A duplicate injection of a sample extract may be performed to display instrument precision.

9.4.1.1. The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference. Report all results and flag outliers.

9.4.2. Isotope dilution analyte recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any isotope dilution analyte is less than 10:1.

9.5. Surrogate/Clean Up Internal Standard

A surrogate compound may be spiked into all air media samples prior to collection. For all other matrices, a clean up internal standard is spiked following extraction and just prior to cleanup, in order to monitor relative loss of isotope dilution analyte during both extraction and cleanup.

9.6. Isotope Dilution Analytes

9.6.1. Isotope dilution analytes must be spiked into all samples, QC samples, and included in all calibrations.

9.6.2. For each sample and QC aliquot, calculate the percent recovery. The percent recovery should be between 40 percent and 135 percent for all nine isotope dilution analytes.

9.6.3. A low or high percent recovery for a blank does not require discarding the analytical data but it may indicate a potential problem with future analytical data. Isotope dilution analyte recoveries are flagged if they are outside the

recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any isotope dilution analyte is less than 10:1.

9.7. Recommended Corrective Actions and Troubleshooting Steps

- Verify satisfactory instrument performance.
- If possible, verify that no error was made while weighing the sample portions.
- Review the analytical procedures with the performing laboratory personnel.

10. CALIBRATION

Calibration and Standardization requires a check of mass resolution (tuning), a check of chromatographic resolution, a verification of switching times (i.e. descriptors), and a calibration curve verification.

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 "Calibration Curves (General)".

10.2. Tuning (Mass Resolution Check)

10.2.1. The mass spectrometer must be operated in the electron ionization mode. A static resolving power of at least 10,000 (10 percent valley definition) must be demonstrated at appropriate masses before any analysis is performed. Corrective actions must be implemented whenever the resolving power does not meet the requirement.

10.2.2. Chromatography time for PCDDs and PCDFs exceeds the long-term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, a mass-drift correction is mandatory. To that effect, it is recommended to select a lock-mass ion from the reference compound (PFK is recommended) used for tuning the mass spectrometer. The selection of the lock-mass ion is dependent on the masses of the ions monitored within each descriptor. Table 6 offers some suggestions for the lock-mass ions. However, an acceptable lock-mass ion at any mass between the lightest and heaviest ion in each descriptor can be used to monitor and correct mass drifts. The level of the reference compound (PFK) metered into the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the most intense selected lock-mass ion signal (regardless of the descriptor number) does not exceed 10 percent of the full-scale deflection for a given set of detector parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

NOTE: Excessive PFK (or any other reference substance) may cause noise problems and contamination of the ion source resulting in downtime for source cleaning.

10.2.3. By using a PFK molecular leak, tune the instrument to meet minimum required resolving power of 10,000 (10 percent valley) at m/z 304.9824 (PFK) or any other reference signal close to m/z 303.9016 (from TCDF). Verify that the exact mass of m/z 380.9760 (PFK) is within 5 ppm of the required value. Note that the selection of the low- and high-mass ions must be such that they provide the largest voltage jump performed in any of the five mass descriptors (Table 6).

10.2.4. Documentation of the instrument resolving power must then be accomplished by recording the peak profile of the high-mass reference signal (m/z 380.9760). The minimum resolving power of 10,000 must be demonstrated on the high-mass ion while it is transmitted at a lower accelerating voltage than the low-mass reference ion, which is transmitted at full sensitivity. The format of the peak profile representation (Figure 3) must allow manual determination of the resolution, i.e., the horizontal axis must be a calibrated mass scale (amu or ppm per division). The result of the peak width measurement (performed at 5 percent of the maximum, which corresponds to the 10-percent valley definition) must appear on the hard copy and cannot exceed 100 ppm at m/z 380.9760 (or 0.038 amu at that particular mass).

10.3. Performance Checks

10.3.1. At the beginning of each 12-hour period during which samples are to be analyzed, aliquots of the 1) GC column performance check solution and 2) high-resolution concentration calibration solution No. 4 (HRCC-4) shall be analyzed to demonstrate adequate GC resolution and sensitivity, response factor reproducibility, and mass range calibration, and to establish the PCDD/PCDF retention time windows. (Note: A HRCC-3 or HRCC-5 may be acquired to meet the requirement of #2 above. This is to provide documentation of consistency for varying concentration levels, and to meet NELAC requirements). A mass resolution check shall also be performed to demonstrate adequate mass resolution using an appropriate reference compound (PFK is recommended). If the required criteria are not met, remedial action must be taken before any samples are analyzed. The mass resolution check will be taken at the beginning and completion of an analytical sequence. An analytical sequence may contain one or more 12 hour periods.

10.3.1.1. Method blanks or solvent blanks are used to demonstrate that the analytical system is free of contamination after the analysis of calibration standards or high level samples. The blank must demonstrate that the system has returned to appropriate background levels prior to continued analysis.

10.3.2. At a minimum, the ions listed in Table 6 for each of the five SIM descriptors

must be monitored. Note that the PeCDF masses (M+2 & M+4) are also monitored in the first descriptor. This is because the first PeCDF isomer elutes closely to the final tetra isomer. The selection (Table 6) of the molecular ions M and M+2 for ^{13}C-HxCDF and ^{13}C-HpCDF rather than M+2 and M+4 (for consistency) is to eliminate, even under high-resolution mass spectrometric conditions, interferences occurring in these two ion channels for samples containing high levels of native HxCDDs and HpCDDs. It is important to maintain the same set of ions for both calibration and sample extract analyses. The recommended mass spectrometer tuning conditions are based on the groups of monitored ions shown in Table 6.

10.3.2.1. The GC column performance check mixture, high-resolution concentration calibration solutions, and the sample fortification solutions may be obtained from the EMSL-CIN. However, if not available from the EMSL-CIN, standards can be obtained from other sources, and solutions can be prepared in the laboratory. Concentrations of all solutions containing 2,3,7,8-substituted native PCDDs/PCDFs, must be verified by comparison with second-source standard solutions.

10.4. Initial Calibration

Initial calibration is required before any samples are analyzed for PCDDs and PCDFs. Initial calibration is also required if any routine calibration (Section 10.5) does not meet the required criteria listed in Section 10.6.

10.4.1. Five high-resolution concentration calibration solutions, listed in Table 5, must be used for the initial calibration.

10.4.2. Tune the instrument with PFK.

10.4.3. Inject 1 or 2 µL of the GC column performance check solution and acquire SIM mass spectral data as described earlier in Section 6.1.3. The total cycle time must be ≤ 1 second. This is analyzed prior to a calibration curve to set descriptor windows only and may not otherwise be documented. The laboratory must not analyze samples until it is demonstrated and documented that the criterion listed in Section 13.1 is met.

10.4.3.1. Select the injection volume based upon the expected target analyte concentration, or expected matrix interferences.

10.4.3.2. The same injection volume must be used for all samples, QC, and standards.

10.4.4. By using the same GC and mass spectrometer conditions that produced acceptable results with the column performance check solution, analyze a 1 or

2-µL portion of each of the five concentration calibration solutions once with the following mass spectrometer operating parameter.

10.4.4.1. The total cycle time for data acquisition must be < 1 second. The total cycle time includes the sum of all dwell times and voltage reset times.

10.4.4.2. Acquire SIM data for all the ions listed in the five descriptors of Table 6.

10.4.4.3. The ratio of integrated ion current for the ions appearing in Table 9 (homologous series quantification ions) must be within the indicated control limits (set for each homologous series).

10.4.4.4. The ratio of integrated ion current for the ions belonging to the ^{13}C labeled isotope dilution analytes and internal standards must be within the control limits stipulated in Table 9.

NOTE: Section 10.4.3 requires that ion ratios be within the specified control limits simultaneously in one run. It is the laboratory's responsibility to take corrective action if the ion abundance ratios are outside the limits.

10.4.5. For each SICP and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the signal-to-noise ratio (S/N) must be better than or equal to 10. This measurement is suggested for any GC peak that has an apparent S/N of less than 5:1. The result of the calculation must appear on the SICP above the GC peak in question.

10.4.5.1. Referring to Table 5, calculate the 17 relative response factors (RRF) for unlabeled target analytes [RRF(n); n=1 to 17] relative to their appropriate isotope dilution analytes (Table 5) and the nine RRFs for the labeled ^{13}C isotope dilution analytes [RRF(m); m=18 to 26] relative to the two internal standards according to the following formulae:

$$RRF(n) = \frac{A_x \times Q_{IDA}}{Q_x \times A_{IDA}} \qquad RRF(m) = \frac{A_{IDA} \times Q_{IS}}{Q_{IDA} \times A_{IS}}$$

Where:

A_x = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 5) for unlabeled PCDDs/PCDFs,

A_{IDA} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 5) for the labeled isotope dilution analytes,

A_{IS} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for the labeled internal standards,

Q_{IDA} = quantity of the isotope dilution analyte injected (pg),

Q_{IS} = quantity of the internal standard injected (pg), and

Q_x = quantity of the unlabeled PCDD/PCDF analyte injected (pg).

The RRF (n) and RRF (m) are dimensionless quantities; the units used to express Q_{IDA}, Q_{IS}, and Qx must be the same.

10.4.5.2. Calculate the RRF(n)s and their respective percent relative standard deviations (%RSD) for the five calibration solutions:

$$\overline{RRF}(n) = (1/5)\sum_{j=1}^{5} RRF_j(n)$$

Where n represents a particular PCDD/PCDF (2,3,7,8-substituted) congener (n = 1 to 17; Table 5), and j is the injection number (or calibration solution number; j = 1 to 5).

10.4.5.3. The relative response factors to be used for the determination of the concentration of total isomers in a homologous series are calculated as follows:

10.4.5.3.1. For congeners that belong to a homologous series containing only one isomer (e.g., OCDD and OCDF) or only one 2,3,7,8-substituted isomer (Table 4; TCDD, PeCDD, HpCDD, and TCDF), the mean RRF used will be the same as the mean RRF determined in Section 10.3.5.2.

NOTE: The calibration solutions do not contain ^{13}C-OCDF as an isotope dilution analyte. This is because a minimum resolving power of 12,000 is required to resolve the [M+6]+ ion of ^{13}C-OCDF from the [M+2]+ ion of OCDD (and [M+4]+ from ^{13}C-OCDF with [M]+ of OCDD). Therefore, the RRF for OCDF is calculated relative to ^{13}C-OCDD.

10.4.5.3.2. For congeners that belong to a homologous series containing more than one 2,3,7,8-substituted isomer (Table 4), the mean RRF used for those homologous series will be the mean of the RRFs calculated for all individual 2,3,7,8-substituted congeners using the equation below:

$$\overline{RRF}(k) = (1/t)\sum_{n=1}^{t} RRF_n$$

Where:

k = 27 to 30, with 27 = PeCDF;

28 = HxCDF; 29 = HxCDD; and 30 = HpCDF,

t = total number of 2,3,7,8-substituted isomers present in the calibration solutions (Table 5) for each homologous series (e.g., two for PeCDF, four for HxCDF, three for HxCDD, two for HpCDF).

NOTE: Presumably, the HRGC/HRMS response factors of different isomers within a homologous series are different. However, this analytical protocol will make the assumption that the HRGC/HRMS responses of all isomers in a homologous series that do not have the 2,3,7,8-substitution patterns are the same as the responses of one or more of the 2,3,7,8-substituted isomer(s) in that homologous series.

10.4.5.4. Relative response factors [RRF(m)] to be used for the determination of the percent recoveries for the nine isotope dilution analytes are calculated as follows:

$$RRF(m) = \frac{A^m_{IDA} \times Q_{IS}}{Q^m_{IDA} \times A_{IS}}$$

$$\overline{RRF}(m) = (1/5)\sum_{j=1}^{5} RRF_j(m)$$

Where:

m =	18 to 26 (congener type)
j =	1 to 5 (injection number),
A_{IDA}^m =	sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given isotope dilution analyte (m = 18 to 26),
A_{IDA} =	sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given isotope dilution analyte (m = 18 to 26),
Q_{IDA} & Q_{IDA}^m =	quantities of, respectively, the internal standard (rs) and a particular isotope dilution analyte (m) injected (pg),
RRF(m) =	relative response factor of a particular isotope dilution analyte (m) relative to an appropriate internal standard, as determined from one injection, and
$\overline{RRF(m)}$ =	calculated mean relative response factor of a particular isotope dilution analyte, as determined from the five initial calibration injections (j).

10.5. Criteria for acceptable calibration

The criteria listed below for acceptable calibration must be met before sample analysis is performed.

10.5.1. The percent relative standard deviations for the mean response factors [RRF(n) and RRF(m)] from the 17 unlabeled standards must be ≤ 20 percent, and those for the nine labeled reference compounds must be ≤ 30 percent.

Note: If Method 8290A criteria are required for the project then both the percent standard relative standard deviation for the mean response factors for the 17 unlabeled standards and the nine labeled reference compounds must be ≤ 20 percent.

10.5.2. The signal/noise ratio (S/N) for the GC signals present in every SICP (including the ones for the labeled standards) must be ≥ 10.

10.5.3. The isotopic ratios (Table 9) must be within the specified control limits.

NOTE: If the criterion for acceptable calibration listed in Section 10.4.1 is met, the analyte-specific RRF can then be considered independent of the analyte quantity for the calibration concentration range. The mean RRFs will be used for all calculations until the routine calibration criteria (Section 10.6) are no longer met. At such time, new mean RRFs will be calculated from a new set of injections of the calibration solutions.

10.6. Routine Calibration (continuing calibration check)

Routine calibrations must be performed at the beginning of (following a successful tune and GC column performance check) and after a 12 hour period. The routine calibration initiates the 12 hour clock during which samples may be subsequently analyzed. The last sample in the sequence must be injected within 12 hours of the routine calibration, followed by the analysis of a closing calibration check. An acceptable closing calibration check standard may be used to initiate the next 12 hour analysis sequence when consecutive acquisition sequences occur. The ending mass resolution check shall be performed after the closing calibration check of an analysis acquisition sequence or after the final bracketing standard when consecutive 12 hour acquisition sequences are run.

10.6.1. Inject 1 or 2 µL of the concentration calibration solution HRCC-4 containing 10 pg/µL of tetrachlorinated congeners, 50 pg/µL of penta-, hexa-, and heptachlorinated congeners, 100 pg/µL of octachlorinated congeners, and the respective isotope dilution analyte and internal standards (Table 5). By using the same HRGC/HRMS conditions as used in Sections 6.1.3 through 6.2, determine and document an acceptable calibration as provided in Section 10.6.

10.7. Criteria for Acceptable Routine Calibration

The following criteria must be met before further analysis is performed. If these criteria are not met, corrective action must be taken, including recalibration if needed.

10.7.1. The measured RRFs [RRF(n)] for the unlabeled standards obtained during the opening continuing calibration must be ± 20 percent of the mean values established during the initial calibration (Section 10.3.5.)

10.7.1.1. The bracketing continuing calibration must be ± 20% of the average RRF calculated from the initial calibration.

10.7.1.1.1. If the target compounds in the ending standard are less than or equal to ± 20% of the average RRF from the initial calibration, the RRFs of the initial calibration shall be used to quantitate the unlabeled isomers.

10.7.1.1.2. If the target analytes are greater than ± 20% but less or equal to ±25% and the samples are non-detect, the data is acceptable and this anomaly is documented. If these isomers are greater than ± 20% but less or equal to ±25% and are positive, an average RRF of the initial and ending daily standard is calculated and used to quantitate the concentration of the affected congener, and the anomaly is documented.

10.7.1.1.3. If the percent deviation of unlabeled compounds exceeds ± 25%, a new initial calibration is initiated within 2 hours following the analysis of the samples. Otherwise, reanalyze all sample extracts with positives for the failed target compounds.

10.7.2. The measured RRFs [RRF(m)] for the labeled standards obtained during the opening continuing calibration must be less than or equal to ± 30 percent of the mean values established during the initial calibration (Section 10.1.5).

10.7.2.1. The bracketing continuing calibration must be ± 30% of the average RRF calculated from the initial calibration.

10.7.2.1.1. If the labelled compounds in the ending standard are less than or equal to ±30% of the average RRF from the initial calibration, the RRFs of the initial calibration shall be used to quantitate the labeled isomers.

10.7.2.1.2. If the isotope dilution analyte analytes are greater than ± 30% but less or equal to ±35%, an average RRF of the initial and ending daily standards is calculated and used to quantitate the concentration of the affected congener.

10.7.2.1.3. If the percent deviation of labeled compounds exceeds ± 35%, reanalyze samples if adversely impacted.

10.7.3. The ion-abundance ratios (Table 9) must be within the allowed control limits.

10.7.4. If either criteria in Sections 10.7.1 or 10.7.2 are not met, additional samples may not be analyzed. Sample data collected must be evaluated for usability. Narrate any reported data from the analytical sequence. If the ion-abundance ratio criterion is not satisfied, refer to the note in Section 10.4.3 for resolution.

10.7.5. If the above criteria (Section 10.7) cannot be satisfied, the entire initial calibration process (Section 10.4) must be repeated.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Sample Dilution Procedure – Simple Dilutions

Dilutions from 2X to 50X can be achieved without respiking the final extract. The calculation to determine the final extract concentration is as follows:

(Concentration of the original extract) x (amount of aliquot taken) x (volume of diluted extract) = final concentration of dilution.

Ex: 50X dilution of original 10 g/20 µL sample

(10 g/20 µL) x (2 µL aliquot + 98 µL keeper) = 1 g/100 µL FV

Record the final sample concentration on the extract label.

11.3. Sample Dilution Procedure – Complex Dilutions

Complex dilution requiring respiking of IDA and IS: Dilutions greater than 50x must be done by diluting and respiking the extract with IDA's and IS. This procedure may require serial dilution to be performed. If this procedure is done, then the sample size must be adjusted to reflect the aliquot taken.

Ex. 100X dilution (original sample with 10 g/20 µL final volume)

Take a 2 µL aliquot (1/10 of original sample) and add 18 µL of solvent keeper. Take a 2 µL aliquot of the dilution (1/100 of the original sample), respike with 1 mL IDA and 20 µL IS, reduced to 20 µL FV.

Record the final sample concentration of the extract label.

11.4. Analytical Procedures

11.4.1. Inject a 1 or 2 µL aliquot of the extract into the GC, operated under the conditions previously used (Section 6.2) to produce acceptable results with the performance check solution.

11.4.2. Acquire SIM data according to Section 6.1.3. Use the same acquisition and mass spectrometer operating conditions previously used to determine the relative response factors (Section 10). Ions characteristic for polychlorinated diphenyl ethers are included in the descriptors listed in Table 6. Their presence is used to monitor their interference during the characterization of PCDFs.

12. CALCULATIONS/DATA REDUCTION

12.1. Identification Criteria

For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet all of the following criteria:

12.1.1. Retention Times

12.1.1.1. For 2,3,7,8-substituted congeners, which have an isotopically labeled isotope dilution analyte or internal standard present in the sample extract, the retention time (at maximum peak height) of the sample components (i.e., the two ions used for quantitation purposes listed in Table 6) must be within -1 and +3 seconds of the retention time of the peak for the isotopically labeled isotope dilution analyte or internal standard at m/z corresponding to the first characteristic ion (of the set of two; Table 6) to obtain a positive identification of these nine 2,3,7,8-substituted PCDDs/PCDFs and OCDD.

12.1.1.2. For 2,3,7,8-substituted compounds that do not have an isotopically labeled isotope dilution analyte present in the sample extract, the relative retention time (relative to the appropriate isotope dilution analyte) must fall within 0.005 relative retention time units of the relative retention times measured in the daily routine calibration. Identification of OCDF is based on its retention time relative to ^{13}C-OCDD as determined from the daily routine calibration results.

12.1.1.3. For non-2,3,7,8-substituted compounds (tetra through octa; totaling 119 congeners), the retention time must be within the corresponding homologous retention time windows established by analyzing the column performance check solution.

12.1.1.4. The ion current responses for both ions used for quantitative purposes (e.g., for TCDDs: m/z 319.8965 and 321.8936) must reach a maximum simultaneously (± 2 seconds).

12.1.1.5. The ion current responses for both ions used for the labeled standards (e.g., for ^{13}C-TCDD: m/z 331.9368 and m/z 333.9339) must reach a maximum simultaneously (± 2 seconds).

12.1.2. Ion Abundance Ratios

The integrated ion current for the two ions used for quantitation purposes must have a ratio between the lower and upper limits established for the homologous series to which the peak is assigned. See Table 9.

12.1.3. Signal-To-Noise Ratio

All ion current intensities must be >2.5 times noise level for positive identification of the PCDD/PCDF compound or a group of coeluting isomers. Figure 4 describes the procedure to be followed for the determination of the S/N.

12.1.4. Polychlorinated Diphenyl Ether Interferences

In addition to the above criteria, the identification of a GC peak as a PCDF can only be made if no signal having a S/N >2.5 is detected, at the same retention time (± 2 seconds), in the corresponding polychlorinated diphenyl ether (PCDPE, Table 6) channel.

12.2. For gas chromatographic peaks that have met the criteria outlined above, calculate the concentration of the PCDD or PCDF compounds using the formula:

$$C_x = \frac{A_x \times Q_{IDA}}{A_{IDA} \times W \times RRF(n)}$$

Where:

C_x = concentration of unlabeled PCDD/PCDF congeners (or group of coeluting isomers within an homologous series) usually in pg/g or pg/L,

Ax = sum of the integrated ion abundances of the quantitation ions (Table 6) for the unlabeled PCDD/PCDFs,

A_{IDA} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled isotope dilution analytes,

Q_{IDA} = quantity, in pg, of the isotope dilution analyte added to the sample before extraction,

W = sample size in g (if solid) or L (if liquid).

$RRF(n)$ = Calculated mean relative response factor for the analyte [RRF(n) with n = 1 to 17; Section 10.3.5].

If the analyte is identified as one of the 2,3,7,8-substituted PCDDs or PCDFs, RRF(n) is the value calculated using the equation in Section 10.3.5.1. However, if it is a non-2,3,7,8-substituted congener, the RRF(k) value is the one calculated using the equation in Section 10.3.5.3.2 [RRF(k) with k = 27 to 30].

12.3. Calculate the percent recovery of the nine isotope dilution analytes measured in the sample extract, using the formula:

$$\text{Isotope Dilution Analytes Percent Recovery} = \frac{A_{IDA} \times Q_{IS}}{Q_{IDA} \times A_{IS} \times RRF(m)} \times 100$$

Where:

A_{IDA} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled isotope dilution analytes,

A_{IS} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standard; the selection of the internal standard depends on the type of congeners (see Table 5, footnotes),

Q_{IDA} = Quantity, in pg, of the isotope dilution analyte added to the sample before extraction,

Q_{IS} = Quantity, in pg, of the internal standard added to the cleaned-up sample residue before HRGC/HRMS analysis, and

$RRF(m)$ = calculated mean relative response factor for the labeled isotope dilution analyte relative to the appropriate (see Table 5, footnotes) internal standard. This represents the mean obtained in Section 10.3.5.4 [RRF(m) with m = 18 to 26].

12.4. If the concentration in the final extract of any of the fifteen 2,3,7,8-substituted PCDD/PCDF compounds (Table 3) exceeds the upper method calibration limit (MCL) for that compound listed in Table 1, the linear range of response versus concentration may have been exceeded. In such cases, the following corrective actions will be undertaken:

12.4.1. If the signal for the analyte has saturated the detector, a single dilution and reanalysis of the extract will be made in an attempt to bring the signal within the range of the detector. If the measured concentration of the analyte is still above the MCL, the reported concentration for the analyte will be qualified appropriately. Some programs, such as DOD QSM, require all compounds to be within the linear calibration range in which a serial dilution must be performed to achieve acceptable quantitation.

12.4.2. If the signal for the analyte is above the MCL but does not saturate the detector, the concentration will be reported and qualified appropriately. Some programs, such as DOD QSM, require all compounds to be within the linear calibration range in which a serial dilution must be performed to achieve acceptable quantitation.

12.5. In either case, **with the approval of the client**, the sample may be re-extracted and/or re-analyzed with one or more of the following adjustments made to the analytical procedure in order to provide a concentration which meets client-specific data quality objectives.

12.5.1. Extraction and analysis of a one tenth aliquot. This is appropriate if it will provide analyte concentration within the MCL and a representative sample aliquot.

12.5.2. Extraction of an aliquot large enough to be representative with an increased concentration of isotope dilution analyte and surrogate spike components added prior to the extraction. The extract is then diluted either prior to or after the cleanup procedures.

12.5.3. Dilution of the original extract. Isotope dilution analyte components are re-spiked at an appropriate level prior to analysis. In this case, the isotope dilution analyte recoveries are taken from the original analysis.

12.6. For the other congeners (including OCDD and OCDF), however, report the measured concentration and indicate that the value exceeds the upper calibration standard.

12.7. The total concentration for each homologous series of PCDD and PCDF is calculated by summing up the concentrations of all positively identified isomers of each homologous series. Therefore, the total should also include the 2,3,7,8-substituted congeners. The total number of GC signals included in the homologous total concentration value may be specified in the report.

12.8. Sample-Specific Estimated Detection Limit

The sample-specific estimated detection limit (EDL) or estimated quantiation limit (EQL, 8290A) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. An EDL/EQL is calculated for each 2,3,7,8-substituted congener that is not identified, regardless of whether or not other non-2,3,7,8-substituted isomers are present. Two methods of calculation can be used, as follows, depending on the type of response produced during the analysis of a particular sample.

12.8.1. Samples giving a response for both quantitation ions (Tables 6 and 9) that is less than 2.5 times the background level.

Use the expression for EDL/EQL (specific 2,3,7,8-substituted PCDD/PCDF) below to calculate an EDL/EQL for each absent 2,3,7,8-substituted PCDD/PCDF (i.e., S/N <2.5). The background level is determined by measuring the range of the noise (peak to peak) for the two quantitation ions (Table 6) of a particular 2,3,7,8-substituted isomer within an homologous series, in the region of the SICP trace corresponding to the elution of the isotope dilution analyte (if the congener possesses an isotope dilution analyte) or in the region of the SICP where the congener is expected to elute by comparison with the routine calibration data (for those congeners that do not have a ^{13}C-labeled standard), multiplying that noise height by 2.5, and relating the product to an estimated concentration that would produce that product height.

NOTE: The quantitation ions for both the unlabeled PCDDs/PCDFs and their isotope dilution analyte must be consistently paired (using either both lighter mass ions or both heavier mass ions).

Use the formula:

$$EDL_{Specific\,2,3,7,8\text{-}subst.PCDD/PCDF} = \frac{2.5 \times H_x \times Q_{IDA}}{H_{IDA} \times W \times RRF(n)}$$

Where:

EDL = estimated detection limit for homologous 2,3,7,8-substituted PCDDs/PCDFs. (also EQL for Method 8290A)

H_x = height of the average noise for one of the quantitation ions (Table 6) for the unlabeled PCDDs/PCDFs.

H_{IDA} = height of one of the quantitation ions (Table 6) for the labeled isotope dilution analytes.

W, RRF (n), and Q_{IDA} retain the same meanings as defined in Section 12.2

12.8.2. Samples characterized by a response above the background level with a S/N of at least 2.5 for at least one of the quantitation ions (Tables 6 and 9).

When the response of a signal having the same retention times as a 2,3,7,8-substituted congener has a S/N in excess of 2.5 and does not meet any of the other qualitative identification criteria listed in Section 12.1, calculate the "Estimated Maximum Possible Concentration" (EMPC) according to the expression shown in Section 12.1, except that Ax in Section 12.1 should represent the sum of the area under the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio. Alternatively, an EDLEQL can be calculated using the above formula and the height of one of the ions as appropriate.

12.9. The relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{|S_1 - S_2|}{(S_1 + S_2)/2} \times 100$$

S_1 and S_2 represent sample and duplicate sample results.

12.10. The 2,3,7,8-TCDD toxic equivalents (TEQ) of PCDDs and PCDFs present in the sample are calculated at the data user's request. This method assigns a 2,3,7,8-TCDD toxicity equivalency factor (TEF) to each of the seventeen 2,3,7,8-substituted PCDDs and PCDFs (Table 10). The 2,3,7,8-TCDD equivalent of the PCDDs and PCDFs present in the sample is calculated by summing the TEF times their concentration for each of the compounds or groups of compounds listed in Table 10.

12.11. Two-GC Column TEF Determination

12.11.1. The concentration of 2,3,7,8-TCDD (see note below), is calculated from the analysis of the sample extract on the 60m DB-5 fused silica capillary column. The chromatographic separation of this isomer must be $\leq$ 25% valley.

12.11.2. For samples that have a positive result for 2,3,7,8-TCDF on the DB-5 column, the extract is reanalyzed on a 30m DB-225 fused silica column. The GC/MS conditions are altered so that only the first descriptor (Table 6) is used. The reported concentration for 2,3,7,8-TCDF is then the result above the lower calibration limit is calculated from the DB-225 analysis. The chromatographic separation between 2,3,7,8-TCDF and any other unlabeled TCDF isomers must be < 25% valley using the column performance check solution for the DB-225 column. Concentration calculations are performed as in Section 12.1 through 12.6.

12.11.3. A DB-225 column can be used in the quantitative analysis of 2,3,7,8-TCDF and 2,3,7,8-TCDD analytes. Since the DB-225 cannot resolve 2,3,7,8-TCDD any positively identified 2,3,7,8-TCDD which exceeds the reporting limit shall be confirmed on a DB-5 column.

12.11.4. For a gas chromatographic peak to be identified as a 2,3,7,8-substituted PCDD/PCDF congener, it must meet the ion abundance (Section 11.5.4) and signal-to-noise ratio criteria. In addition, the retention time identification criterion described in Section 11.5.4 applies here for congeners for which a carbon-labeled analog is available in the sample extract. However, the relative retention time (RRT) of the 2,3,7,8-substituted congeners for which no carbon-labeled analogs are available must fall within 0.006 units of the carbon-labeled standard RRT. Experimentally, this is accomplished by using the attributions described in Table 11 and the results from the routine

calibration run on the DB-5 column.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

It must be documented that all applicable system performance criteria specified were met before analysis of any sample is performed. Table 7 provides recommended GC conditions that can be used to satisfy the required criteria. A GC column performance check is only required at the beginning of each 12-hour period during which samples are analyzed.

13.5. GC Column Performance

13.5.1. Inject 1 or 2 µL of the column performance check solution and acquire selected ion monitoring (SIM) data as described in Section 6.1.3 within a total cycle time of < 1 second.

13.5.2. The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other TCDD isomers must be resolved with a valley of $\leq$ 25

percent (Figure 2),

Where:

$$\text{Valley Percent} = \left(\frac{x}{y}\right) \times 100$$

x = measured as in Figure 2 from the 2,3,7,8-closest TCDD eluting isomer,

y = the peak height of 2,3,7,8-TCDD

13.5.3. It is the responsibility of the laboratory to verify the conditions suitable for the appropriate resolution of 2,3,7,8-TCDD from all other TCDD isomers. The GC column performance check solution also contains the known first and last PCDD/PCDF eluters under the conditions specified in this protocol. Their retention times are used for qualitative and quantitative purposes. The peak for 2,3,7,8-TCDD must be labeled on the chromatograms. The chromatograms showing the first and last eluters of a homologous series must be included.

13.5.4. The retention times for the switching of SIM ions characteristic of one homologous series to the next higher homologous series must be indicated in the SICP. Accurate switching at the appropriate times is absolutely necessary for accurate monitoring of these compounds.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Autovials containing assorted solvents and extracts. As the autovials are removed from the instrument after analysis, they are collected in archive boxes and retained pending additional instructions. When no longer needed, the archive boxes are moved to the waste disposal area for disposal as PCB waste.

16. REFERENCES/CROSS REFERENCES

16.1. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 8290 Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry September 1994.

16.2. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update IV. Method 8290A Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry February 2007.

16.3. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 0023A, Sampling Method for Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans Emissions from Stationary Sources. December 1996.

16.4. Compendium Method TO-9A "Determination of Polychlorinated, Polybrominated, and Brominated, Cholorinated Dibenxo-p-dioxins and Dibenzofurans in Ambient Air", EPA compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, second edition, January 1997.

16.5. Protocol for the Analysis of 2,3,7,8-TCDD by HRGC/HRMS". J. S. Stanley and T. M. Sack, EPA 600/4-86-004.

16.6. "Safety in Academic Chemistry Laboratories", American Chemical Society Publication, Committee on Chemical Safety (3rd Edition, 1979.)

16.7. "Carcinogens - Working with Carcinogens". Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control. National Institute for Occupational Safety and Health. Publication No. 77-206, August 1977.

16.8. "OSHA Safety and Health Standards, General Industry", (29 CFR 1910) Occupational Safety and Health Administration, OSHA 2206 (revised January 1976).

17. METHOD MODIFICATIONS

17.1. Modifications from EPA 8290 and EPA 8290A

17.1.1. The methods specify that 2 µL injections are used throughout the analysis. If an instrument demonstrates adequate sensitivity and chromatographic resolution, then the analyst may use 1 µL injections for all performance checks, standards, QC samples, and samples.

17.1.2. In Section 2.7 of Method 8290 and 8290A, a retention time window of 0.005 RT units is used to tentatively identify unlabeled PCDD/PCDFs for which there are no corresponding labeled isotope dilution analytes. All available labeled isotope dilution analytes are used; therefore, a retention time window

of -1 to +3 seconds is used to identify all compounds. See Section 7.8.4.1 of Method 8290 and 7.9 of Method 8290A.

17.1.3. Tetradecane instead of nonane is used as the final solvent to increase the stability of extracts and standards. Tetradecane is less volatile than nonane. Loss of analyte as a result of solvent incompatibility is monitored through recovery checks and calibration acceptance criteria.

17.2. Modifications from TO-9A method

17.2.1. The ^{37}Cl-2,3,7,8-TCDD surrogate is present at varying levels in the calibration curve (0.5-200 pg/ µL).

17.2.2. The laboratory uses 2 labeled internal standards for the quantitation of labeled isotope dilution analytes.

17.2.3. The final volume is adjusted to 20 µL in tetradecane.

17.2.4. Calibration and quantitation are performed in accordance to this SOP.

18. ATTACHMENTS

18.1. Table 1 - Types of Matrices

18.2. Table 2 - Composition of the Sample Fortification and Internal Standard Solutions.

18.3. Table 3 - The Fifteen 2,3,7,8-Substituted PCDD and PCDF Congeners

18.4. Table 4 - Isomers of Chlorinated Dioxins and Furans

18.5. Table 5 - Concentrations of Calibration Solutions

18.6. Table 6 - Ions Monitored for PCDDs/PCDFs

18.7. Table 7 - Recommended GC Operating Conditions

18.8. Table 8 - Congeners in the GC Performance Evaluation Solution (DB-5)

18.9. Table 9 - Theoretical Ion Abundance Ratios and Control Limits

18.10. Table 10 - 2,3,7,8-TCDD Equivalent Factors

18.11. Table 11 - TEF: Analyte Relative Retention Time Reference Attributes

18.12. Figure 1 - Compound Structure

18.13. Figure 2 - GC Performance Check Chromatogram on the DB-5 Column

18.14. Figure 3 - PFK Peak Profile

18.15. Figure 4 - Manual Determination of Signal-to-Noise

18.16. Appendix A - Periodic Wipe Test Performance

19. REVISION HISTORY

19.1. WS-ID-0005, Revision 7.5, Effective 04/19/2013

19.1.1. Replaced all instances of 'internal standard' with isotope dilution analyte' and all instances of 'recovery standard' with 'internal standard' to conform with TALS naming guidelines.

19.1.2. Editorial revisions.

19.2. WS-ID-0005, Revision 7.4, Effective 01/14/2011.

19.2.1. Editorial revisions.

19.3. WS-ID-0005, Revision 7.3, Effective 12/30/2009

19.3.1. Editorial revisions.

19.4. WS-ID-0005, Revision 7.2, Effective 11/02/2009

19.4.1. Section 6.1: Inserted "Preventive and routine maintenance is described in the 'Schedule of Routine Maintenance' in the QAM."

19.4.2. Section 12.1.2: Removed the word "presumptive" and inserted "above the lower calibration limit" after the word result.

TABLE 1

Types of Matrices, Sample Sizes and 2,3,7,8-TCDD-Based Method Calibration Limits (Parts per Trillion)

	Water	**Soil Sediment Paper Pulp**	**Fly Ash**	**Human/ Fish Tissue**	**Adipose Tissue**	**Sludges, Fuel Oil**	**Still-Bottom**	**Ambient or Source Samples**
Lower MCL(a)	0.01	1.0	2.0	1.0	2.0	10	20	40
Upper MCL(a)	4.0	400	400	400	400	2000	4000	8000
Weight (g)	1000	10	10	10	10	2.0	1.0	1 sample
IDA Spiking Levels (ng)	2.0	2.0	2.0	2.0	2.0	2.0	2.0	4.0
Final Extract Volume (µL)	20	20	20	20	20	20	20	20

(a) For other congeners, multiply the values by 1 for TCDF, by 5 for PeCDD/PeCDF/HxCDD/HxCDF/HpCDD/HpCDF, and by 10 for OCDD/OCDF.

TABLE 2

Composition of the Sample Fortification and Internal Standard Solutions

Analyte	Sample Fortification Solution Concentration pg/μL; Solvent: Isooctane	Internal Standard Solution Concentration pg/μL; Solvent: Tetradecane
^{13}C-2,3,7,8-TCDD	2[a], 100[c]	--
^{13}C -2,3,7,8-TCDF	2[a], 100[c]	--
^{13}C -1,2,3,4-TCDD	--	100
^{13}C -1,2,3,7,8-PeCDD	2[a], 100[c]	--
^{13}C -1,2,3,7,8-PeCDF	2[a], 100[c]	--
^{13}C -1,2,3,6,7,8-HxCDD	2[a], 100[c]	--
^{13}C -1,2,3,4,7,8-HxCDF[d]	2[a], 100[c]	--
^{13}C -1,2,3,7,8,9-HxCDD	--	100
^{37}Cl-2,3,7,8-TCDD[b][c]	0.8[b], 100[c]	
	100[c]	
^{13}C -2,3,4,7,8-PeCDF[c]	100[c]	
^{13}C -1,2,3,6,7,8-HxCDF[c][d]	100[c]	
^{13}C -1,2,3,4,7,8-HxCDD[c]	100[c]	
^{13}C -1,2,3,4,7,8,9-HpCDD[c]	100[c]	
^{13}C -1,2,3,4,6,7,8-HpCDD	2[a], 100[c]	--
^{13}C -1,2,3,4,6,7,8-HpCDF	2[a], 100[c]	--
^{13}C -OCDD	4[a], 200[c]	--

(a) Standard 8290, 8290A, Method 23, Method 0023A, TO9 and TO9A Sample Fortification Solution concentrations
(b) Method TO9 and TO9A surrogate concentrations
(c) Method 23 and Method 0023A surrogate concentrations
(d) ^{13}C-1,2,3,6,7,8-HxCDF is used as a Sample Fortification Solution and ^{13}C -1,2,3,4,7,8-HxCDF is used as a surrogate solution in Method 0023A

TABLE 3

The Seventeen 2,3,7,8-Substituted PCDD and PCDF Congeners

PCDD	PCDF
2,3,7,8-TCDD(*)	2,3,7,8-TCDF(*)
1,2,3,7,8-PeCDD(*)	1,2,3,7,8-PeCDD(*)
1,2,3,6,7,8-HxCDD(*)	2,3,4,7,8-PeCDF
1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDD(+)	1,2,3,7,8,9-HxCDF
1,2,3,4,6,7,8-HpCDD(*)	1,2,3,4,7,8-HxCDF(*)
1,2,3,4,5,6,7,8-OCDD(*)	2,3,4,6,7,8-HxCDF
	1,2,3,4,6,7,8-HpCDF(*)
	1,2,3,4,7,8,9-HpCDF
	1,2,3,4,5,6,7,8-OCDF

(*)The ^{13}C -labeled analog is used as an isotope dilution analyte.
(+)The ^{13}C -labeled analog is used as a internal standard.

TABLE 4

Isomers of Chlorinated Dioxins and Furans as a Function of the Number of Chlorine Atoms

# of Chlorine Atoms	# of Dioxin Isomers	# of 2,3,7,8 Isomers	# of Furan Isomers	# of 2,3,7,8 Isomers
1	2	---	4	---
2	10	---	16	---
3	14	---	28	---
4	22	1	38	1
5	14	1	28	2
6	10	3	16	4
7	2	1	4	2
8	1	1	1	1
Total	**75**	**7**	**135**	**10**

TABLE 5

High Resolution Concentration Calibration Solutions

	Compound	Concentration (ng/mL)				
RRF (n)(m)		**CS2**	**CS3**	**CS4 (ICV(6))**	**CS5**	**CS6**
	Native CDDs and CDFs					
1	2,3,7,8-TCDD	0.5	2	10	40	200
2	2,3,7,8-TCDF	0.5	2	10	40	200
3	1,2,3,7,8-PeCDD	2.5	10	50	200	1000
4	1,2,3,7,8-PeCDF	2.5	10	50	200	1000
5	2,3,4,7,8-PeCDF	2.5	10	50	200	1000
6	1,2,3,4,7,8-HxCDD	2.5	10	50	200	1000
7	1,2,3,6,7,8-HxCDD	2.5	10	50	200	1000
8	1,2,3,7,8,9-HxCDD	2.5	10	50	200	1000
9	1,2,3,4,7,8-HxCDF	2.5	10	50	200	1000
10	1,2,3,6,7,8-HxCDF	2.5	10	50	200	1000
11	1,2,3,7,8,9-HxCDF	2.5	10	50	200	1000
12	2,3,4,6,7,8-HxCDF	2.5	10	50	200	1000
13	1,2,3,4,6,7,8-HpCDD	2.5	10	50	200	1000
14	1,2,3,4,6,7,8-HpCDF	2.5	10	50	200	1000
15	1,2,3,4,7,8,9-HpCDF	2.5	10	50	200	1000
16	OCDD	5.0	20	100	400	2000
17	OCDF	5.0	20	100	400	2000
	Labeled CDDs and CDFs					
18	$^{13}C_{12}$-2,3,7,8-TCDD	100	100	100	100	100
19	$^{13}C_{12}$-2,3,7,8-TCDF	100	100	100	100	100
20	$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	100	100	100	100
21	$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	100	100	100	100
	$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	100	100	100	100
22	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	100	100	100	100
23	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	100	100	100	100
	$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	100	100	100	100
24	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	100	100	100	100
25	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,4,7,8,9-	100	100	100	100	100

	Compound	**Concentration (ng/mL)**				
RRF (n)(m)		**CS2**	**CS3**	**CS4 (ICV(6))**	**CS5**	**CS6**
	HpCDF					
26	$^{13}C_{12}$-OCDD	200	200	200	200	200
	Cleanup Standard/ FS					
	$^{37}Cl_4$-2,3,7,8-TCDD	0.5	2	10	40	200
	Internal Standards					
	$^{13}C_{12}$-1,2,3,4-TCDD	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	100	100	100	100	100

TABLE 6*
Elemental Compositions and Exact Masses of the Ions
Monitored by HR/MS for PCDD's and PCDF's

Descriptor	Exact m/z [1]	m/z Type	Elemental Composition	Substance [2]
1	292.9825	QC	C_7F_{11}	PFK
	303.9016	M	$C_{12}H_4{}^{35}Cl_4O$	TCDF
	305.8987	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF
	315.9419	M	$^{13}C_{12}H_4{}^{35}Cl_4O$	TCDF [3]
	317.9389	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF [3]
	319.8965	M	$C_{12}H_4{}^{35}Cl_4O_2$	TCDD
	321.8936	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD
	327.8847	M	$C_{12}H_4{}^{37}Cl_4O_2$	TCDD [4]
	330.9792	Lock	C_7F_{13}	PFK
	331.9368	M	$^{13}C_{12}H_4{}^{35}Cl_4O_2$	TCDD [3]
	333.9339	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD [3]
	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	341.8567	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}ClO$	PeCDF
	375.8364	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}ClO$	HxCDPE
	409.7974	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}ClO$	HpCDPE
2	330.9792	QC	C_7F_{13}	PFK
	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	341.8567	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF
	342.9792	Lock	C_8F_{12}	PFK
	351.9000	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	353.8970	M+4	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF [3]
	354.9792	Lock	C_9F_{13}	PFK
	355.8546	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD
	357.8516	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD
	366.9793	QC	C_9F_{13}	PFK
	367.8949	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD [3]
	369.8919	M+4	$^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD [3]
	409.7974	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}ClO$	HpCDPE
3	373.8208	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF
	375.8178	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$	HxCDF
	380.9760	Lock	C_8F_{15}	PFK
	383.8639	M	$^{13}C_{12}H_2{}^{35}Cl_6O$	HxCDF [3]
	385.8610	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF [3]
	389.8157	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD
	391.8127	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD
	392.9760	Lock	C_9F_{15}	PFK
	401.8559	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD [3]
	403.8529	M+4	$^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD [3]
	430.9728	QC	C_9F_{17}	PFK
	445.7550	M+4	$C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$	OCDPE
4	392.9760	QC	C_9F_{15}	PFK
	407.7818	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF
	409.7789	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O$	HpCDF

Descriptor	Exact m/z [1]	m/z Type	Elemental Composition	Substance [2]
	417.8253	M	$^{13}C_{12}H^{35}Cl_7O$	HpCDF [3]
	419.8220	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF [3]
	423.7766	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD
	425.7737	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD
	430.9729	Lock	C_9F_{17}	PFK
	435.8169	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD [3]
	437.8140	M+4	$^{13}C_{12}H^{35}Cl_5{}^{37}CL_2O_2$	HpCDD [3]
	479.7165	M+4	$C_{12}H^{35}Cl_7{}^{37}Cl_2O$	NCDPE
5	392.9760	QC	C_9F_{15}	PFK
	441.7428	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO$	OCDF
	442.9728	Lock	$C_{10}F_{17}$	PFK
	443.7399	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O$	OCDF
	457.7377	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD
	459.7348	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD
	469.7779	M+2	$^{13}C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD [3]
	471.7750	M+4	$^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD [3]
	479.7165	M+4	$C_{12}Cl_8{}^{37}Cl_2O$	NCDPE
	513.6775	M+4	$^{13}C_{12}{}^{35}Cl_8{}^{37}Cl_2O$	DCDPE

---

[a] The following nuclidic masses were used:

H =	1.007825	O =	15.994915
C =	12.000000	^{35}Cl =	34.968853
^{13}C =	13.003355	^{37}Cl =	36.965903
F =	18.9984		

S = Isotope dilution analyte/internal standard

*The homologous groups for functions 1-3 do not use the same lockmass as described in Table 6. They use masses 316.9824, 366.9792, and 380.9760, respectively.

TABLE 7

Recommended GC Operating Conditions

The GC Operating Conditions (Temperatures (°C), and Times (minutes)) Are as Follows:

Injector Temperature: 280°C
Interface Temperature: 280°C
Initial Temperature and Time: 190°C / 1 Minute

Temperature Program: 190°C, increasing at a rate of 4°C per minute up to 240°C, and maintaining at this temperature until the last tetra of the tetra- group has eluted from the column. (The total time required for this is approximately 25 minutes, depending on the length of the column). The maintained temperature of 240°C is then increased to 320°C at the rate of 20°C per minute and held at this level until the last compound (octa-group) has eluted from the column.

TABLE 8

PCDD and PCDF Congeners Present in the GC Performance Evaluation Solution and Used for Defining the Homologous GC Retention Time Windows on a 60-M DB-5 Column[(b)]

# of Chlorine Atoms	PCDD Positional Isomer		PCDF Positional Isomer	
	Early Eluter	Late Eluter	Early Eluter	Late Eluter
4[(a)]	1,3,6,8	1,2,8,9	1,3,6,8	1,2,8,9
5	1,2,4,6,8/1,2,4,7,9	1,2,3,8,9	1,3,4,6,8	1,2,3,8,9
6	1,2,3,4,6,8	1,2,3,4,6,7	1,2,3,4,6,8	1,2,3,4,8,9
7	1,2,3,4,6,7,8	1,2,3,4,6,7,9	1,2,3,4,6,7,8	1,2,3,4,6,7,9
8	1,2,3,4,6,7,8,9		1,2,3,4,6,7,8,9	

[(a)] In addition to these two PCDD isomers, the 1,2,3,4-, 1,2,3,7-, 1,2,3,8-, 2,3,7,8-, $^{13}C_{12}$-2,3,7,8-, and 1,2,3,9-TCDD isomers must also be present.

(b) The PCDF Congeners present in GC the Performance Evaluation Solution for the 30 m DB-225 column include:

- 1,2,3,9-TCDF
- 2,3,7,8-TCDF
- 2,3,4,7-TCDF
- $^{13}C_{12}$-2,3,7,8-TCDF

Column performance criteria is met when the percent valleys between the 2,3,7,8-TCDF analyte and the closest eluting isomers are $\leq$ 25%.

TABLE 9

Theoretical Ion Abundance Ratios and Their Control Limits for PCDDs and PCDFs

# of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits	
			Lower	Upper
4	M / M+2	0.77	0.65	0.89
5	M+2 / M+4	1.55	1.32	1.78
6	M+2 / M+4	1.24	1.05	1.43
6[(a)]	M / M+2	0.51	0.43	0.59
7[(b)]	M / M+2	0.44	0.37	0.51
7	M+2 / M+4	1.04	0.88	1.20
8	M+2 / M+4	0.89	0.76	1.02

[(a)] Used only for ^{13}C-HxCDF (IS) [(b)] Used only for ^{13}C-HpCDF (IS)

TABLE 10

2,3,7,8-TCDD Equivalent Factors (TEFs) for the Polychlorinated Dibenzodioxins and Dibenzofurans

Number	Compound(s)	TEF
1	2,3,7,8-TCDD	1.00
2	1,2,3,7,8-PeCDD	0.50
3	1,2,3,6,7,8-HxCDD	0.10
4	1,2,3,7,8,9-HxCDD	0.10
5	1,2,3,4,7,8-HxCDD	0.10
6	1,2,3,4,6,7,8-HpCDD	0.01
7	OCDD	0.001
8	2,3,6,7-TCDF	0.1
9	1,2,3,7,8-PeCDF	0.05
10	2,3,4,7,8PeCDF	0.5
11	1,2,3,6,7,8-HxCDF	0.1
12	1,2,3,7,8,9-HxCDF	0.1
13	1,2,3,4,7,8-HxCDF	0.1
14	2,3,4,6,7,8-HxCDF	0.1
15	1,2,3,4,6,7,8-HpCDF	0.01
16	1,2,3,4,7,8,9-HpCDF	0.01
17	OCDF	0.001

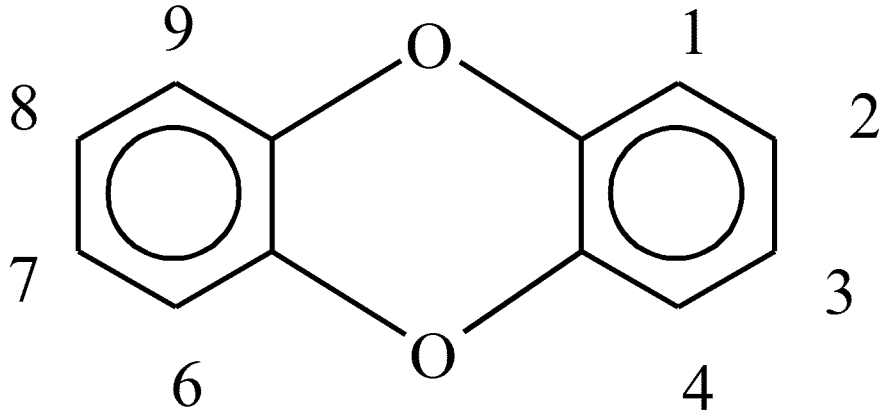
TABLE 11

Toxicity Equivalency Factor:
Analyte Relative Retention Time Reference Attributes

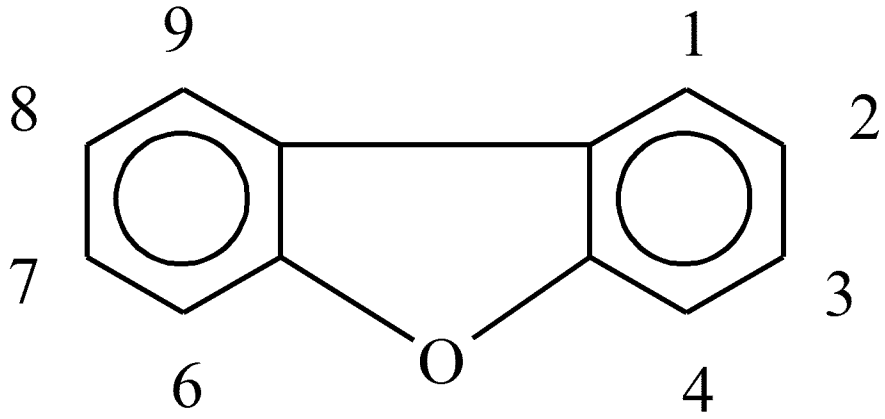
Analyte	Analyte RRT Reference (a)
1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF
1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF
2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF

(a) The retention time of 2,3,4,7,8-PeCDF on the DB-5 column is measured relative to $^{13}C_{12}$-1,3,7,8-PeCDF and the retention time of 1,2,3,4,7,8,9-HpCDF relative to $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF

FIGURE 1
Structure of Dibenzodioxin and Dibenzofuran



Dibenzodioxin



Dibenzofuran

FIGURE 2

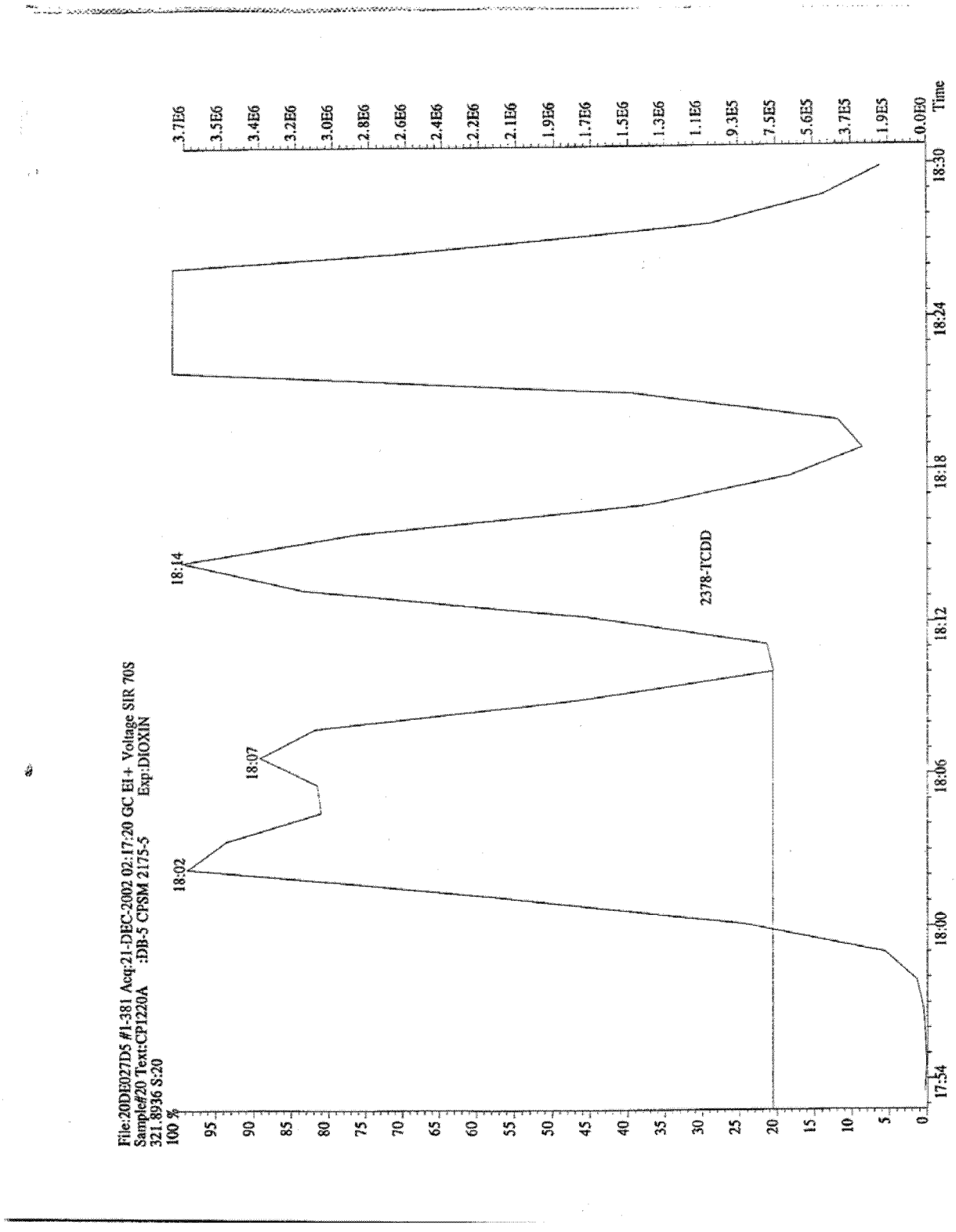

File:20DE027D5 #1-381 Acq:21-DEC-2002 02:17:20 GC EI+ Voltage SIR 70S
Sample#20 Text:CP1220A :DB-5 CPSM 2175-5 Exp:DIOXIN
321.8936 S:20
100 %
18:02
18:07
18:14
2378-TCDD
Time
17:54
18:00
18:06
18:12
18:18
18:24
18:30
0.0E0
1.9E5
3.7E5
5.6E5
7.5E5
9.3E5
1.1E6
1.3E6
1.5E6
1.7E6
1.9E6
2.1E6
2.2E6
2.4E6
2.6E6
2.8E6
3.0E6
3.2E6
3.4E6
3.5E6
3.7E6

Figure 3

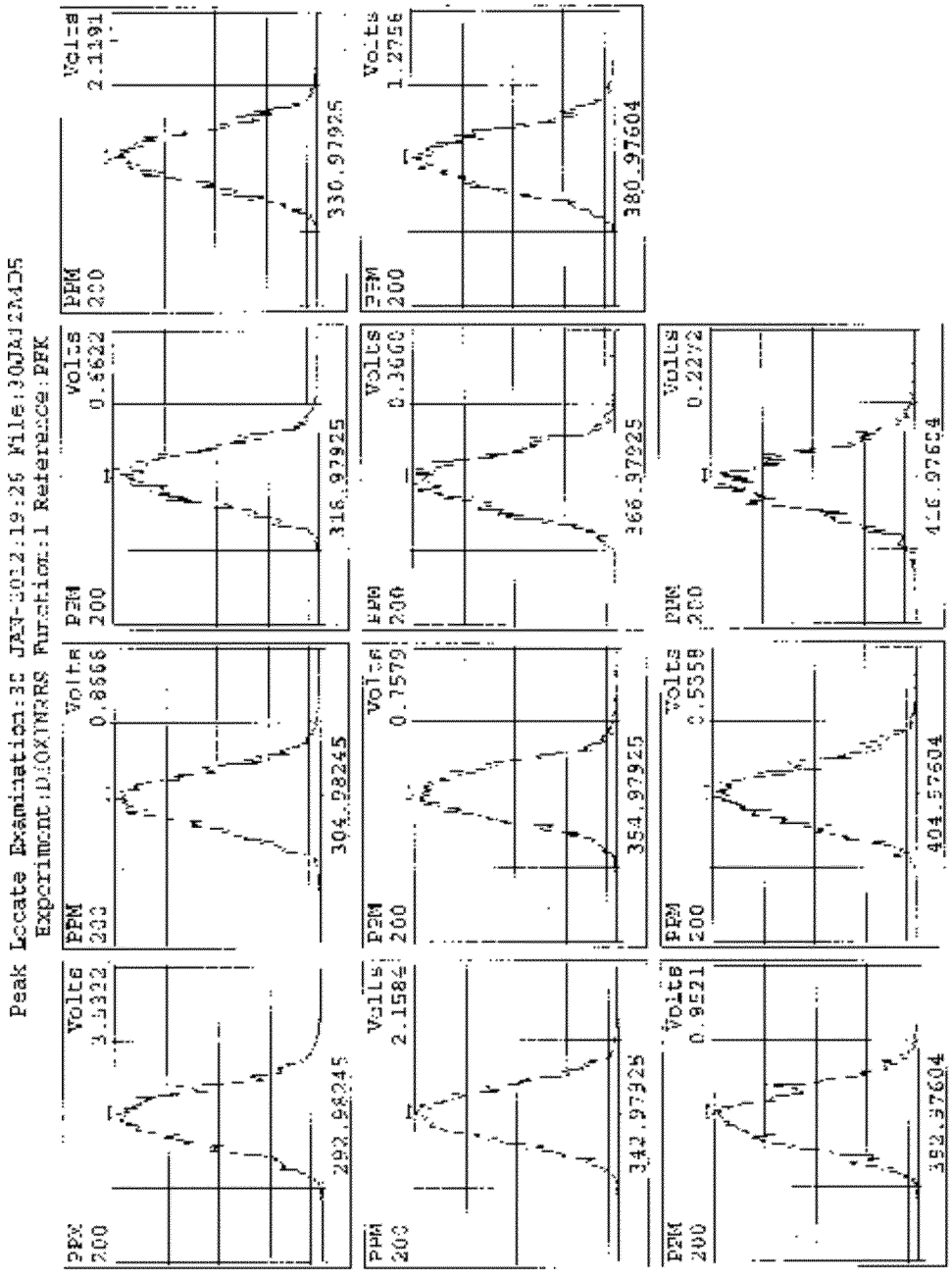
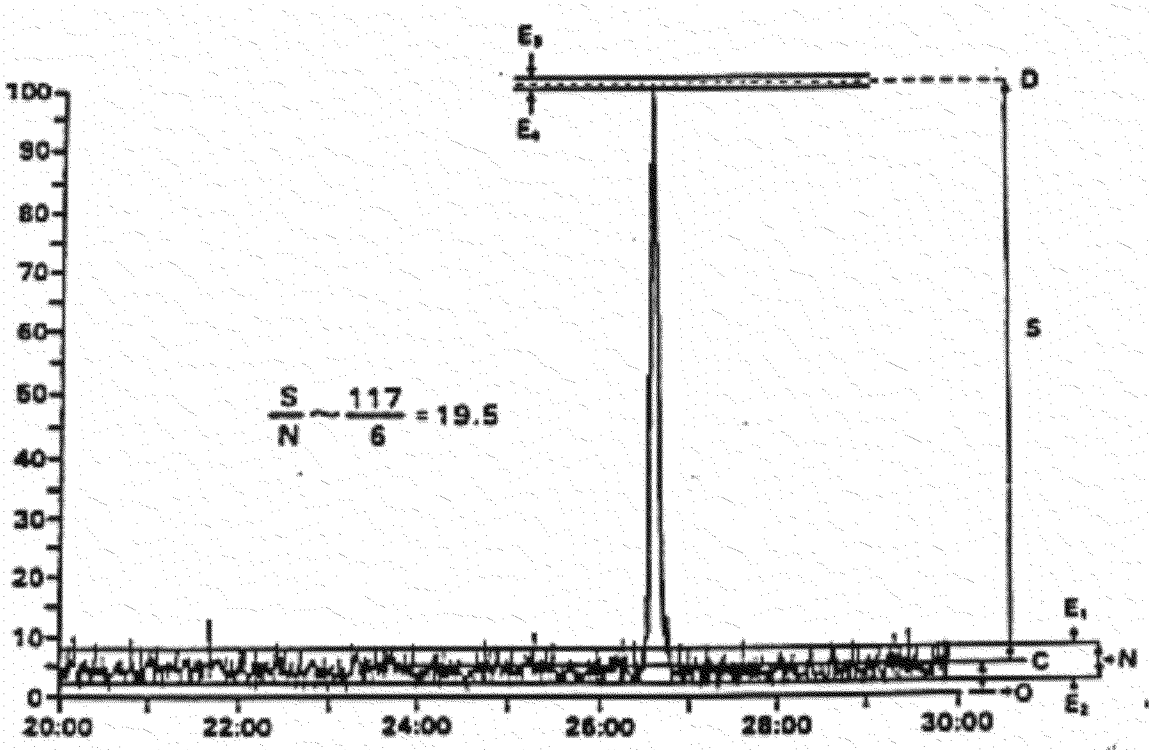

Peak Locate Examination:3S JAN-2012:19:26 File:30JAN2A05
Experiment:DIOXINARS Purification:1 Reference:PFK
PPM
200
Volts
3.9322
292.98245
Volts
2.1584
342.97925
PPM
200
Volts
0.8666
304.98245
Volts
0.7579
354.97925
PPM
200
Volts
0.5622
316.97925
Volts
0.3600
366.97925
PPM
200
Volts
2.1191
330.97925
Volts
1.2756
380.97604
PPM
200
Volts
0.9521
392.97604
Volts
0.5358
404.97604
PPM
200
Volts
0.2272
416.97654
PPM
200

FIGURE 4



Manual determination of S/N.

The peak height (S) is measured between the mean noise (lines C and D). These mean signal values are obtained by tracing the line between the baseline average noise extremes, E1 and E2, and between the apex average noise extremes, E3 and E4, at the apex of the signal.

NOTE: It is imperative that the instrument interface amplifier electronic zero offset be set high enough so that negative going baseline noise is recorded.

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November 1992

APPENDIX A

This procedure is designed for the periodic evaluation of potential contamination by 2,3,7,8-substituted PCDD/PCDF congeners of the working areas inside the laboratory.

PERFORMING WIPE TEST

Perform the wipe tests on surface areas of two inches by one foot with laboratory wipers saturated with distilled-in-glass acetone or appropriate solvent using a pair of clean stainless steel forceps. Use one wiper for each of the designated areas. Combine the wipers to one composite sample in an extraction jar containing 200 mL distilled-in-glass hexane. Place an equal number of unused wipers in 200 mL hexane and use this as a control.

SAMPLE PREPARATION

Close the jar containing the wipes and 200 mL hexane and extract for 20 minutes using a wrist-action shaker. Use an appropriate means to reduce the volume to approximately 1.0 mL. Put through an alumina column to clean up potential interfering compounds. Add appropriate amount of internal standard.

EXTRACT ANALYSIS

Concentrate the contents of the vial to a final volume of 20 µL (either in a minivial or in a capillary tube). Inject 2 µL of each extract (wipe and control) onto a capillary column and analyze for 2,3,7,8-substituted PCDDs/PCDFs as specified in the analytical method Section 11 (this exhibit). Perform calculations according to Section 12 (this exhibit).

REPORTING FORMAT

Report the presence of 2,3,7,8-substituted PCDDs and PCDFs as a quantity (pg or ng) per wipe test experiment (WTE). Under the conditions outlined in this analytical protocol, a lower limit of calibration of 25 pg/WTE is expected for 2,3,7,8-TCDD. A positive response for the blank (control) is defined as a signal in the TCDD retention time window at any of the masses monitored which is equivalent to or above 8 pg of 2,3,7,8-TCDD per WTE. For other congeners, use the multiplication factors listed in Table 1, footnote (a) (e.g., for OCDD, the lower MCL is 25 x 5 = 125 pg/WTE and the positive response for the blank would be 8 x 5 = 40 pg). Also, report the recoveries of the isotope dilution analytes during the simplified cleanup procedure.

FREQUENCY OF WIPE TESTS

Wipe tests should be performed when there is evidence of contamination in the method blanks.

CORRECTIVE ACTION

An upper limit of 25 pg per TCDD isomer and per wipe test experiment is allowed. (Use multiplication factors listed in footnote (a) from Table 1 for other congeners.) This value corresponds to the lower calibration limit of the analytical method. Steps to correct the contamination must be taken whenever these levels are exceeded. To that effect, first vacuum the working places (hoods, benches, sink) using a vacuum cleaner equipped with a high-efficiency particulate absorbent (HEPA) filter and then wash with a detergent. A new set of wipes should be analyzed before anyone is allowed to work in the dioxin area of the laboratory.

The test results and the decontamination procedure must be reviewed with EH&S.

Controlled Copy
Copy No. unc

SOP No: KNOX-MS-0001
Revision No: 14
Revision Date: 07/16/13
Implementation Date: 08/16/13

TESTAMERICA KNOXVILLE

STANDARD OPERATING PROCEDURE

TITLE: VOA CANISTER ANALYSIS

(SUPERSEDES: KNOX-MS-0001, Revision 13)

Prepared By: _____

Reviewed By: _____ 7/22/13
Technical Specialist

Approved By: _____ 7/22/13
Quality Assurance Manager

Approved By: _____ 7-22-13
Environmental, Health and Safety Coordinator

Approved By: _____ 07/22/13
Laboratory Director

1. Scope and Application

1.1. The purpose of this standard operating procedure is to define the procedures and quality control necessary to analyze samples collected in SUMMA®, TO-Can®, SilcoCan® or similarly passivated stainless steel canisters.

1.2. This procedure is applicable to the analysis of ambient air, indoor air, landfill gases, soil gases, vapor intrusion, and other gaseous samples. It is based on EPA Methods TO-14A and TO-15.

1.2.1. See Appendix I for the list of target analytes and reporting limits.

1.3. Responsibilities to perform this procedure in the lab are as follows:

Position	Responsibilities
Analyst	- Prepares and analyzes samples - Summarizes/assembles data package - Reviews the data package
Team/Group Leader	- Schedules/assigns analyses - Reviews data package

2. Summary of Method

2.1. Microscale Purge and Trap (MSPT): A precisely measured aliquot is removed from the canister or Tedlar bag and concentrated on a cryogenic trap (typically a glass bead trap). The cryogenic trap is desorbed. Polar and nonpolar compounds are quantitatively transferred to a subambient Tenax™ trap. Most of the water remains on the Cryotrap and CO2 passes through the Tenax™ trap and is vented. The Tenax™ trap is thermally desorbed to the on-column cryofocuser. Sample components are separated by temperature programmed gas chromatography and detected with a quadrupole mass spectrometer

2.2. Cold Trap Dehydration (CTD): Allows for direct trapping of the sample onto a Tenax trap in Module 2, with dehydration of the sample occurring in Module 1 (a "blank" trap coated with a highly inert surface treatment). By cooling M1 down to subambient temperatures, most of the water is removed, while PPB and sub-PPB compounds remain in the gas phase for direct trapping onto the M2 Tenax™ trap at subambient temperature. After collecting the requested volume of standard/sample, both the M1 and M2 trap are flushed with helium to remove any remaining air. As a precaution to any target compound losses in M1, the M1 trap is then heated to 10C and a forward purge of helium is used to recover any target compounds that may have condensed in M1 for quantitative transfer to M2. M3 is then cooled to cryogenic temperature, allowing M2 to be back

desorbed and its contents focused onto a pre-column for rapid and splitless injection into a GC or GCMS. Remaining water in the sample should be as little as 0.2-0.3ul, which is handled by the GCMS better, where the water will spread out and its impact on the mass spectrometer reduced.

2.3. The compounds analyzed by this method are listed in Appendix I, Table 1.

3. Definitions

3.1. Canister - a stainless steel container, typically 6-liter volume, equipped with a stainless steel shut-off valve, suitable for use from vacuum to 40 psig. 1-L cans are available for reduced volume analysis.

3.2. SUMMA® Passivation - a proprietary treatment process used to deactivate stainless steel surfaces. It produces a pure chrome/nickel oxide surface that features a high level of inertness.

3.3. Absolute pressure - pressure measured with reference to absolute zero pressure, expressed as kpa, mmHg, or psia.

3.4. Gauge pressure - pressure above atmospheric pressure as measured by a standard gauge. Zero gauge pressure is equal to ambient atmospheric pressure, expressed as mmHg, inches Hg, or psig.

3.5. Polar compound - Oxygen-containing compound capable of forming hydrogen bonds in water; compound having significant solubility in water.

3.6. Batch – A batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same 24 hour time period. The Quality Control batch must contain a blank and a Laboratory Control Sample (LCS). Refer to the QC Program document (QA-003, current revision) for further details of the batch definition.

3.7. Additional definitions can be found in the TestAmerica Knoxville QAM glossary.

3.8. Tedlar bag - Tedlar bags are manufactured from PVF (Tedlar) film with a polypropylene valve and septum. Various volume capacities available.

4. Interferences

4.1. Only compounds having both a similar mass spectrum and GC retention time would be expected to interfere in the method. The most common occurrence of this would be with structural isomers.

4.2. Large concentrations of water, methane, or carbon dioxide may limit the size of the aliquot that can be effectively cryotrapped. This may elevate the quantitation limits obtainable for samples of this type.

4.3. Matrix interferences may be caused by non-target contaminants that are present in the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination, or evaluate the next sample for blank acceptance criteria. The autosampler and concentrator may require extensive bake-out and cleaning after a high-level sample.

5. Safety

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2. Procedures are carried out in a manner that protects the health and safety of all associates. Exposure to chemicals and samples will be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made. The preparation of all standards, reagents and glassware cleaning procedures that involve solvents will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.3. All work must be stopped in the event of a known or potential compromise to the health and safety of any associate. The situation must be reported **immediately** to a laboratory supervisor.

5.4. Specific Safety Concerns or Requirements

5.4.1. The effluents of sample splitters for the gas chromatograph and roughing pumps on the mass spectrometer must be vented to the laboratory hood exhaust system or must pass through an activated charcoal filter.

5.4.2. The autosampler, concentrator, gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.4.3. The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.4.4. There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.4.5. Liquid Nitrogen is used to cool traps in the concentrator. The analyst needs to be aware of locations of those zones and warm them to room temperature prior to working on them. The effluent of the traps must be vented to a laboratory hood or outside the room.

5.5. Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Liquid Nitrogen	Hazardous (asphyxiant and cryogenic burns)	n/a (O2 levels should be maintained above 19.5%)	As an asphyxiant, nitrogen can displace oxygen levels below 19.5 %. Individuals breathing in such an atmosphere may experience symptoms which include headaches, ringing in ears, dizziness, drowsiness, unconsciousness, nausea vomiting, and depressing of all the senses. Contact of the liquid with the skin can lead to severe cryogenic burns or dermatitis. Contact of the liquid with the eyes can cause pain, redness, severe cryogenic burns, and prolonged exposure could cause blindness. Contact with the undiluted liquid will cause frostbite, ulceration of the skin (which may be delayed in appearance for several hours), blistering and pain. Contact with rapidly expanding gas poses a frostbite hazard.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetonitrile	Flammable Poison	40 ppm-TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Acetone	Flammable	1000 ppm (TWA)	Inhalation may cause coughing, dizziness, dullness, and headache. Contact causes redness, pain, drying and cracking of the skin. Vapors cause eye irritation. Eye splashes may cause severe irritation, with stinging, tearing, redness and pain.
Benzene	Carcinogen Flammable Poison	1 ppm-TWA 5 ppm-STEL	Toxic by ingestion, inhalation and absorption. Causes headache, nausea, dizziness, weakness and breathing difficulties. This material is irritating on contact with the skin and eyes and may cause permanent eye damage.

Carbon Tetrachloride	Carcinogen Poison	10 ppm-TWA 200 ppm-STEL	Toxic by ingestion, inhalation and absorption. Causes headache, nausea, dizziness and narcosis. Contact with skin or eyes may cause irritation. Consumption of alcohol may increase toxic effects
Chloroform	Carcinogen Irritant	50 ppm Ceiling	Acts as a relatively potent anesthetic. Irritates respiratory tract and causes central nervous system effects, including headache, drowsiness, dizziness. Causes skin irritation resulting in redness and pain. Removes natural oils. May be absorbed through skin. Vapors cause pain and irritation to eyes. Splashes may cause severe irritation and possible eye damage.
1,4-Dichlorobenzene	Irritant	75 ppm-TWA	Can cause irritation by ingestion and inhalation. Causes nausea, vomiting and diarrhea. Contact with material or vapors can cause irritation to skin and eyes.
Vinyl Chloride	Carcinogen Flammable Poison	1 ppm TWA	Toxic by inhalation, ingestion and absorption. Can cause respiratory irritation, dizziness, weakness, fatigue, nausea and headache. Contact with the material can cause eye and skin irritation.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.6. Bulk Nitrogen Usage Procedures - All procedures require full time monitoring at work station and an audible O_2 monitor which must be activated when any liquid nitrogen valve is open.

5.6.1. Filling of portable liquid N_2 Dewar flask:

5.6.1.1. Position face shield on head.

5.6.1.2. Open valve to portable Dewar flask.

5.6.1.3. Close valve when portable Dewar flask has filled and seal lid with gloves.

5.6.1.4. Open lid on portable Dewar flask and fill trap on canister cleaning apparatus with liquid N_2.

5.6.1.5. Remove face shield and gloves.

6. Equipment and Supplies

6.1. Canisters (SUMMA®, TO-Can®, SilcoCan®), 1, 6-, 15-, and 30-liter sizes, preferably equipped with two valves and integral vacuum/pressure gauge, Restek or equivalent.

6.2. Static gas dilution bottles (SGDB), nominally 2000 ml, with mininert valves, Entech Instruments Inc., or equivalent.

6.3. Syringes, gas-tight, 10 uL, 50 uL, 500 uL, 1000 uL, 2.5 mL, 50 mL, 500 mL, all side port needle, Hamilton, Inc., or equivalent.

6.4. Gas Chromatograph/Mass Spectrometer System, Agilent HP 6890 GC and 5973 or 5975 MSD or equivalent.

6.5. Fused silica capillary column, 60 m x 0.32 x 1um film DB-5, J&W Scientific, or equivalent.

6.6. Vacuum pump, Model 726.3 TTP, KNF Newberger, or equivalent.

6.7. Canister concentrator system, Model 7100, 7100A or 7200, Entech Instruments Inc., with a Model 7016CA or 7016D, 16-position auto sampler.

6.8. Gauges: (certified annually):

6.8.1. Digital gauge 0 to 30" Hg vacuum, Dwyer or equivalent

6.8.2. Digital gauge 0 to -29.9" Hg vacuum, 0 to 99.9 psi, Dwyer or equivalent

6.9. Tedlar Bags: Variety of sizes. SKC or equivalent.

6.10. Fisherbrand traceable workstation digital barometer, or equivalent (use within certification date from manufacturer)

6.11. Mass flow controller: McMillan model 80SD-4 or equivalent

6.11.1. The flow of the mass flow controller is checked each day before use. Nitrogen is purged through the flow controller to the flow meter and the system is allowed to stabilize. The flow controller may be adjusted to achieve the desired flow rate, and the reading from the certified flow meter is entered into the mass flow controller logbook. The observed flow through the flow meter is used to calculate the time required to achieve the desired volume. The reading from the mass flow controller is not used.

6.12. Flow meter: Agilent model ADM 3000, Alicat model M-500 SCCM-D or equivalent (NIST-traceable certified annually).

7. Reagents and Standards

7.1. Helium, ultra high purity, 99.999+%, Air Products, or equivalent.

7.2. Liquid nitrogen, Air Products, or equivalent.

7.3. Nitrogen, ultra high purity, Air Products or equivalent

Internal/Surrogate Standard (all at 50 ppb) in nitrogen, 2000 psig, Scott Specialty Gases, or equivalent:

CAS NUMBER	Internal Standards	MOLECULAR WEIGHT (ng/n mole)

74-97-5	bromochloromethane	129.4
540-36-3	1,4-difluorobenzene	114.1
3114-55-4	chlorobenzene-d5	117.6
	Surrogate	
460-00-4	4-bromofluorobenzene	175.0

7.3.1. Since there are no response factor requirements, no surrogate requirements, and no second source requirement for internal standards or surrogates from the reference methods, a 5-year expiration date is assigned from the manufacturer's preparation date.

7.3.2. A sufficient volume from the internal/surrogate standard cylinder is transferred to a canister to produce a positive pressure.

7.3.3. The working internal/surrogate standard may be used as long as the pressure in the canister remains above ambient pressure and is not past its expiration date.

7.3.4. The Entech is programmed to add 40 mL of the internal standard/surrogate can. This results in a concentration of 4 ppb/v of internal standard/surrogate (based on 500 mL volume).

7.4. Primary Target Initial Calibration/Laboratory Control Sample and Initial Calibration Verification Standard (2^{nd} source) Gaseous Standards: target compounds, 1000 ppb v/v, vendor-certified high-pressure aluminum cylinder.

7.4.1. An expiration date of one year from the date of vendor certification is assigned to the standard cylinder. This expiration date may be extended through comparison against an unexpired standard that meets the second source standard criteria in Section 10.4, or recertified by the vendor.

7.4.2. The initial calibration and the initial calibration verification standard (2^{nd} source) are from different lots provided by the manufacturer. See section 10.4 for ICV requirements.

7.5. Additional Standards: Neat materials, not contained in the certified cylinders, can be added to a SGDB either individually or as a mix.

7.5.1. If the desired compound is a gas at room temperature, a measured volume is injected into an evacuated canister and pressurized. See section 12.8 for calculation. If the desired compound is a liquid or solid at room temperature, the volume of each compound to be added to the SGDB is back-calculated to the desired final concentration in the canister. See section 12.5 and 12.6 for calculation.

7.5.2. A cleaned SGDB is heated to approximately 60□C for 24 hours before use. Other SGDB and canister standards are stored at room temperature. If the analytes prove to be plating/condensing in the SGDB at room temperature, then the SGDB is heated to approximately 60□C before use.

7.5.3. Transfer from a SGDB to a canister is performed in a gas-tight syringe equipped with an on-off valve. The syringe is inserted into the SGDB and flushed (primed) several times by filling and evacuating the syringe without withdrawing the syringe from the bottle. The valve is closed after filling the syringe to the desired volume and before withdrawing the syringe from the bottle. The syringe is quickly removed from the SGDB and inserted into a canister capped with a septa. The valves to the syringe and the can are opened allowing the contents of the syringe to transfer. If using a heated syringe, this step must be completed quickly before the syringe cools.

7.6. Primary and Second Source "High" Standard (for a 15-L can; for a 6-L can, reduce the volume of standards appropriately).

7.6.1. 2 mL of reagent water is injected through a septum (inserted into a ¼ in. nut) into a clean evacuated (approx. -29" Hg) 15-L canister. A nitrogen line is attached and opened to blow the water from the valve area into the can, to the desired vacuum/pressure.

7.6.2. 200 ppb(v/v) example: 7.5 liters of a 1000 ppb(v/v) high pressure standard is added to the canister through a mass flow controller (for example, 107 min & 8 seconds at 70 mL/min.). The canister is then pressurized with nitrogen to 2.5 atm for a final volume of 37.5 L and a final concentration of 200 ppb(v/v).

7.7. Working Level Standard Preparation:

7.7.1. 100 uL of reagent water is injected through a septum (inserted into a ¼ in. nut) into a clean evacuated (approx. -29" Hg) 6-L canister. A nitrogen line is attached and opened to blow the water from the valve area into the can to the desired vacuum/pressure.

7.7.2. 20 ppb(v/v) from a 200 ppb(v/v) example: 1.5 L of a 200 ppb(v/v) standard is added to a 6-L canister with a mass flow controller (for example, 21 min & 26 seconds at 70 mL/min.). The canister is pressurized to 2.5 atm for a final volume of 15 L and a final concentration of 20 ppb(v/v).

7.8. Alternate concentrations of the high and working standards may be made as long as the calculations, concentrations and volumes are adjusted appropriately and preparation is clearly documented in the standard preparation logbook.

7.9. Approved SGDB and canister stock standards (section 7.6) may be used for 6 months from the date of preparation or the earliest expiration of parent standard, whichever comes first. Working canister standards (7.7 and 7.8) may be used for two months from the date of preparation or the earliest expiration of parent standard, whichever comes first.

7.10. Mixes and neat compounds (that are not in SGDB, cans, or cylinders) are stored at the manufacturer's recommended storage conditions.

8. Sample Collection, Preservation and Storage

8.1. Sampling is not performed for this method by TestAmerica Knoxville. For information regarding sample shipping, refer to SOP KNOX-SC-0003, Receipt and Log-In of Commercial Samples, current revision.

Container Type	Preservative	Holding Time
Canister	None	30 days from sample collection to sample analysis.
Tedlar bag	None	The analyst must either analyze the sample in the tedlar bag within 72 hours from collection, or transfer the sample from the tedlar bag to a canister within 72 hours from sample collection. If the sample in the tedlar bag is transferred to a canister within 72 hours from collection, the holding time is 30 day from sample collection. Tedlar bags are not listed as sampling containers in the reference methods.

9. Quality Control

9.1. Internal/Surrogate Standards

9.1.1. Internal standards are added to each analytical standard, blank and sample. The acceptance criteria for each internal standard's area for every analysis must be $\pm$ 40% recovery of the internal standard area from the continuing calibration standard. The acceptance criteria for each internal standard's retention time in every analysis must be within $\pm$ 20 seconds (0.33 minutes) of the internal standard retention time from the continuing

calibration standard. See Section 7.4 for preparation of the working internal standard/surrogate standard canister.

9.1.1.1. If the internal standard areas for a sample are outside their limits, the cause is determined. If it is a result of a system problem, then the problem must be corrected and the sample reanalyzed with acceptable results. If it is the result of a matrix effect, the sample must be reanalyzed to confirm this, unless the effect is caused by high levels of target or non-target compounds co-eluting with or interfering with the surrogate or internal standards. If there is not an obvious matrix effect, the sample must be reanalyzed without subsequent dilution unless a dilution is needed to bring target analytes within the instrument calibration range.

9.1.2. The reference methods do not require addition of surrogates. Bromofluorobenzene is used as a surrogate, and recovery must fall within 60% to 140%.

9.1.2.1. Since the concentration of the surrogate is constant in all initial calibration points, the response factor of the surrogates in the daily calibration may be substituted as a one-point calibration to calculate the recoveries in the samples and QC.

9.2. System Blanks (Method Blanks)

9.2.1. For each 24-hour tune in which samples are analyzed or every 20 samples, whichever is more frequent, an acceptable system blank must be analyzed before samples analysis.

9.2.1.1. A system blank is defined as a cleaned canister, humidified with reagent water and filled with UHP nitrogen.

9.2.1.2. Typically, a 30L canister, pressurized to 25-30 psi with humidified nitrogen is used for the blank. A lot check from the can cleaning system can be used as a system blank (See section 9.5).

9.2.1.3. An acceptable system blank is one with all target analytes less than 0.2 ppb/v. The data may still be reported if the concentration of the analyte is less than the laboratory reporting limit (see Table 1), and meets internal standard and surrogate requirements in section 9.1. Any samples associated with a method blank with results above 0.2 ppb/v are flagged in the data report. If a blank has a

reportable result between the RL and the MDL, the associated samples are also flagged with a B qualifier. A blank value above the RL may be accepted if the sample result is greater than 10 X the blank value or not detected above the RL.

9.2.2. If a system blank does not meet the above criteria, then the blank must be reanalyzed or a new blank prepared and analyzed with acceptable results.

9.3. Laboratory Control Standard (LCS)

9.3.1. The LCS is defined as a working standard made by the same method as analytical standards, using the same source materials. It is used to assess analytical control of this procedure. The LCS is analyzed every 24 hour tune or every 20 samples, whichever is more frequent. See Section 7.5, 7.7, 7.8, and 7.9 for details regarding the preparation of the LCS standard canister.

9.3.1.1. The daily calibration verification may also serve as the LCS as long as it meets the criteria of both the LCS and the daily calibration verification.

9.3.2. All target analytes requested are control analytes in the LCS. Sporadic marginal exceedances are allowed where more than 11 analytes are requested. See the table below. The recovery of all target analytes must be within 70-130%, with the marginal exceedence allowance of 60-140% recovery as indicated in Table 1. Provisory analytes must be within 60-140% recovery with the marginal exceedence allowance of 50-150% recovery. See Appendix I Table 1 for the provisory analytes. Naphthalene, n-Dodecane and 1,2,3-Trichlorobenzene have assigned control limits of 40-140%.

Number of target analytes in LCS	Allowable # of marginal exceedances of LCS control limits
>90	5
71 - 90	4
51 - 70	3
31 - 50	2
11 - 30	1
< 11	0

9.3.3. The internal standards and surrogate must pass criteria specified in section 9.1.

9.3.4. An LCS is considered to be "out of control" if any target analyte is outside marginal exceedance limits, or if the total number of marginal exceedances is more than the allowed number.

9.3.5. If marginal exceedances are observed, the analyst must review the previous LCS (e.g., review the control chart) for that instrument for each analyte marginally exceeding the control limits to determine if the marginal exceedance is a consecutive occurrence. If there are two consecutive marginal exceedances for the same analyte, the LCS is considered "out of control" and an NCM must be generated and corrective action taken.

9.3.5.1. When evaluating the control chart, the analyst should also check whether there was more than one out of the last three consecutive LCSs outside control limits. If more than one out of the last three LCSs was outside the LCS control limits but within the marginal exceedance limits, then the analyst should evaluate the system for non-random systematic trends.

9.3.6. Samples analyzed along with an LCS determined to be "out of control" shall be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate data qualifying codes.

9.3.6.1. If the LCS recovery for target analytes are all biased high outside acceptance limits and those target analytes are not detected in any of the associated samples above the reporting limit, the sample data may be reported with qualification in the project narrative.

9.3.6.2. Analytes that are biased high in the LCS and not detected in the associated samples are counted in the total number of allowable marginal exceedances.

9.4. Duplicate Analysis

9.4.1. A duplicate is analyzed with every 20 samples. It is not reported unless specifically requested

9.4.2. Selected samples for duplicates are rotated among client samples, and must not include trip blanks or field blanks.

9.4.3. The acceptance criteria for the duplicate analysis are $\leq$ 25% RPD for target compounds that are greater than 5 times the RL. No criteria for n-butanol. The calculations are given in section 12.18.

9.4.4. If the RPD is outside acceptance criteria for the duplicate, the sample is rerun once. If upon reanalysis, the duplicate does not meet acceptance criteria, the original sample data is qualified in the project narrative.

9.4.5. Due to limited sample volume, duplicates are not performed for Tedlar bags unless otherwise specified in the project requirements.

9.5. Canister Blank Checking

9.5.1. From each cleaned lot of canisters, a canister is selected and pressurized with UHP humidified nitrogen. (See SOP KNOX-MS-0022, current revision, "Canister Cleaning and Preparation").

9.5.2. A blank check is analyzed within 24 hours of a valid tune check and calibration. Alternatively, a calibration at the reporting limit may be used to quantitate results.

9.5.3. Note: If the CCV recovery for a target analyte is biased high outside acceptance limits and that target analyte is not detected in any of the associated samples above the reporting limit, the canister blank check sample data may be reported with qualification in the project narrative if submitted to the client.

9.5.4. A blank check passes if there are no target analytes above the reporting limit, and the internal standards pass criteria in section 9.1. Cans are considered certified "clean" if the result for all analytes are below 0.2 ppb/v. However the can may still be used to collect samples if the concentration of the target analyte is less than the reporting limit. If analytes are detected in the can being certified as clean above 0.2 ppb/v and below the reporting limit, this will be noted on the blank check quantitation report.

9.5.5. If a blank check canister does not pass, the can may be re-analyzed. If the acceptance criteria are still not met, the entire lot of canisters must be re-cleaned, and a blank check from the re-cleaned lot must pass.

9.6. Nitrogen check

9.6.1. Before a new nitrogen cylinder is used for pressurization of samples or standards, it must be analyzed as a blank and pass all the criteria in section 9.2.1.3.

9.7. Annual gauge calibration: The gauges that are used in calculations to measure cylinder and canister pressure or vacuum, and calibration standard flow rates must be certified annually. Digital barometers may be used within the vendor

assigned expiration date, then either calibrated annually or replaced. The mass flow controller that is used to prepare calibration standards is verified before each use with a flow meter that has been certified annually

10. Calibration and Standardization

10.1. Instrument Conditions: The following steps are part of the software's automatic tuning procedure and are performed as needed.

10.1.1. Mass assignments of the mass spectrometer are checked and adjusted using perfluorotributylamine (PFTBA FC43).

10.1.2. The mass spectrometer is tuned to meet the criteria for BFB (see Figure 1a and 1b).

10.1.3. The mass spectrometer is adjusted to minimize noise (see instrument manufacturer instruction manuals).

10.1.4. See Appendix III for examples of GC/MS and GC instrument parameters.

10.2. Daily Tune Check

10.2.1. 50 ng or less of BFB is analyzed for each 24-hour time tune period; the 24-hour time period begins at the moment of injection of BFB. All abundance criteria for BFB in Figure 1a or 1b must be met before the analysis of standards, QC samples or client samples. Figure 1a is the TO-14A method criteria, which is more stringent than the TO-15 method criteria (Figure 1b). The default criteria is the TO-14A tuning criteria (Figure 1a). However if the tune fails TO-14A criteria but meets TO-15 criteria, then samples logged in for TO-15 may be analyzed, but not samples for TO-14A.

10.2.2. The BFB must be acquired in the following manner: Three scans (the apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is conducted using a single scan prior to the elution of BFB.

10.2.3. Once the BFB passes criteria, the same mass spectral conditions used for the BFB must be used to acquire the data in that 24-hour tune period, until the next BFB event.

10.2.4. Correction Action: If the daily tune check does not meet acceptance criteria for the requested method, refer to Section 10.1 and retune the mass spectrometer. Also refer to Section 11.7 for guidance on instrument

troubleshooting. An acceptable daily tune check must be obtained before initial calibration and daily calibration verification.

10.3. Initial Calibration

10.3.1. The GC/MS system must be calibrated with at least 5 concentrations that span the monitoring range of interest. The dynamic range of the curve is generally 0.2 ppb v/v to 40 ppb v/v based on 200 mL sample analysis for normal level reporting limits for most analytes, and 0.08 ppb v/v to 16 ppb v/v based on 500 mL sample analysis. The concentration of the low standard of the calibration must be at or below the reporting limit. If quadratic fit is required, there must be at least 6 points. See Appendix IV for the recommended calibration amounts.

10.3.2. See chart below to obtain the typical desired levels of quantitation. This is a typical schematic of the calibration; however the standard can concentration, calibration levels and calculated concentrations may be different, as long as the calibration rules in 10.3.10 and 10.3.12 are followed. See Appendix IV for the calibration table of analytes. If the actual standard amount trapped is greater than 5% from the programmed volume, the actual volume trapped is documented and used in calculations.

Example 8 standard canister calibration series for 200ml analysis (ppb/v/v)

Standard can concentration (ppbv/v)	0.2	0.2	0.4	1.0	2.5	5	10	20	40
# mLs analyzed	100	200	200	200	200	200	200	200	200
Calculated concentration (ppb v/v)	0.1	0.2	0.4	1.0	2.5	5	10	20	40

10.3.3. See Appendix I, Table 1 for suggested quantitation ions.

10.3.4. A calibration curve is valid for all target analytes if the relative standard deviation (RSD) of the relative response factors is $\leq$30% for each target analyte, with the following allowance: up to two target analytes may have an RSD $\leq$ 40%.

10.3.5. The internal standard area response at each calibration level must be within 40% of the mean area response over the initial calibration range for each internal standard.

10.3.6. The retention time (RT) shift for each of the internal standards at each calibration level must be within 20 seconds of the retention time of the mean calibration for each internal standard.

10.3.7. Each analyte at each level must be within 0.06 RRT units of the mean RRT.

10.3.8. If the curve is acceptable and there is time remaining in the 24-hour tune, blanks, LCS's and samples may be analyzed.

10.3.9. The concentrations in the samples, LCS's and blanks are calculated using the response factors from the initial calibration curve.

10.3.10. Linear or quadratic curve fits may be used. Use of $1/Concentration^2$ weighting may be used to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response. The coefficient of determination (r^2) must be ≥ 0.990.

10.3.11. Analyst may elect to drop points from the calibration to improve subsequent quantitation. The rules for dropping points are:

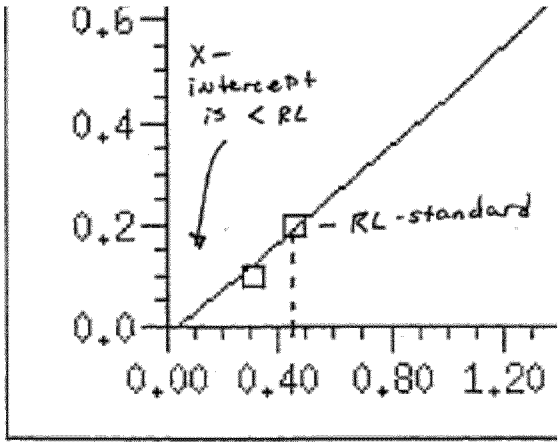
- May drop points below the RL as long as there is a point remaining at or below the RL.
- May drop high points, decreasing linear range.
- May NOT drop a point between points.

For more guidance see "Selection of Calibration Points" Policy CA-T-P-002, current revision.

10.3.12. Rules for curve use:

- The r^2 value obtained from Target must be ≥ 0.990.
- At least 5 points must be used for average or linear curve.
- At least 6 points must be used for a quadratic curve.
- For quadratic curves, the tangent line to the slope of the curve must be continuous and have either only positive or negative slopes (i.e., no parabolas or breaks in the curve). Quadratic curves cannot be used to extend the calibration range. Quadratic curves are only used if the compound has historically exhibited a nonlinear response.
- Forcing through zero is allowed. To activate "force through zero" in Target, select "Force" for "curve origin". "Include" zero for "curve origin" must NOT be used.
- If "forced through zero" is not used, the X and Y-intercept must be below the RL.
- To evaluate the Y-intercept, multiply the positive Y-intercept value by the internal standard amount. The resulting value must be less than the RL.
- Negative Y-intercepts indicate an X-intercept. To evaluate the X-intercept, the intercept from the slope must be less than the intercept of a

vertical line from the reporting limit standard drawn down to the X-intercept. See example below:



10.3.13. The high point calibration standard is checked for saturation. If a quantitation ion saturates the mass spectrometer, the analyte will be removed from the calibration series, and the next highest concentration is checked for saturation as well. Saturation is present when an ion peak in Target reaches a Y axis maximum of 8.4×10^6.

10.3.14. Corrective Action: If the initial calibration fails to meet acceptance criteria a new instrument calibration must be analyzed that meets the acceptance criteria listed in Section 10.3.4. through 10.3.7. Also refer to Section 11.7. for guidance on troubleshooting the instrument. The initial calibration must meet acceptance criteria for all requested analytes prior to sample analysis.

10.4. Initial Calibration Verification (ICV)

10.4.1. The ICV is a second source standard containing all the target compounds in Appendix I, Table 1. The ICV is analyzed after the initial calibration and before any samples are analyzed.

10.4.2. A working standard from an independently prepared stock containing all analytes is also analyzed as the ICV for analytes not included in Table 1.

10.4.3. For each analyte, a percent recovery (%R) is calculated using the response factor from the initial calibration. The ICV is valid for all analytes if the %R is between 65% and 135% for each analyte

10.4.4. Corrective Action: If the ICV fails to meet acceptance criteria a new ICV calibration must be analyzed that meets the acceptance criteria listed in Section 10.4.3. Also refer to Section 11.7. for guidance on troubleshooting

the instrument. The initial calibration verification must meet acceptance criteria for all requested analytes prior to sample analysis.

10.5. Daily Calibration Verification

10.5.1. A mid-level standard is analyzed following the daily tune check (section 10.2) as the calibration verification standard. Typically, this is 100 mL of the 10-ppb/v can. Alternate concentration/volumes may be analyzed.

10.5.2. For all target analytes, a percent difference (%D) or percent drift is calculated using the response from the calibration verification standard and compared to the current initial calibration curve.

10.5.3. A calibration verification standard is acceptable if the %D or % drift is $\leq$30% for all target analytes. However, data may be reported from a calibration verification standard outside 30% as long as the target analytes meet the LCS criteria stated in section 9.3.2. Analytes greater than 30% D in the CCV must be clearly noted in the data report.

10.5.4. The daily calibration verification may also serve as the LCS as long as it meets the criteria of both the LCS (section 9) and the daily calibration verification.

10.5.5. Corrective Action: If the calibration verification standard does not meet the above criteria, corrective action must be taken and/or a new initial calibration performed unless project specific analyte QC criteria are met. Corrective action may include a reanalysis of the calibration verification standard. If reanalysis of the standard does not meet acceptance criteria, further corrective action may include performing instrument maintenance, or preparation of a new working calibration verification standard. Also refer to Section 11.7. for guidance on troubleshooting the instrument. Either of these corrective actions must be followed by successful analysis of the calibration verification standard and reanalysis of any affected samples. If these corrective actions do not result in acceptable calibration verification, a new initial calibration must be performed.

10.5.6. If the recovery for a target analyte is biased high outside acceptance limits and that target analyte is not detected in any of the associated samples above the reporting limit, the sample data may be reported with qualification in the project narrative.

10.5.7. Since the concentration of surrogate is constant in all initial calibration points, the response factor of the surrogates in the daily calibration may be substituted as a one point calibration to calculate the recovery in the samples and QC.

11. Procedure

11.1. Canister Preparation

11.1.1. Use the following guidelines when checking a sample upon receipt:

- Tedlar bags are inspected to ensure that the valve is closed and the bags are not leaking. Bags must be analyzed or transferred to a can within 72 hours of collection. Tedlar bags are analyzed directly from the bag or transferred to an evacuated can within 72 hours of sampling. If the entire bag is transferred to a can, the bag needle valve septum is pierced with a needle attached to a 1-L or a 6-L evacuated can, and the entire contents transferred. If only a portion of the bag is to be transferred, a measured aliquot of the bag is transferred via a clean syringe through a septum attached to the top of a 1-L or a 6-L can. After transfer, the can is then pressurized to a positive pressure with humidified nitrogen and the pressure is recorded. The lab default is to analyze Tedlar bags at a 20x dilution. Based on a default dilution factor at the bench, the RLs and MDLs will be 20 times higher for Tedlar bag analysis. (If a client requests lower RLs than 20x the standard this will need to be communicated to the lab via special instructions.) If a client requests RLs lower than 20x and the client is supplying the tedlar bags, the PM should request that the client send an unused bag to be logged in and analyzed along with their samples as a media blank check. If a client requests RLs lower than 20x and TestAmerica Knoxville is supplying the tedlar bags, the PM should have sample receiving set aside and log in a Tedlar bag from the same lot as a media check.

- For canisters, attach a vacuum/pressure gauge to the top of the can with a line attached to an evacuated cylinder. With the sample can closed, open the valve on the evacuated cylinder to remove air in the line and gauge. Observe that the vacuum is near -27" Hg or lower. If the vacuum is higher, evacuate the cylinder to lower than -27" Hg to prevent the contents of the cylinder from backflushing into the sample can. Close the evacuated cylinder valve and open the sample valve. Observe and record the vacuum/pressure reading of the sample.

- 1-L cans received between -10" Hg vacuum and a positive pressure are ready for a 20 mL analysis. If more volume is expected to be analyzed, the can will have to be pressurized in order to obtain more volume from the can.

- Cans received with a high positive pressure are assumed to have been collected with an active sampler and are analyzed as received.

- Summa canisters are not an active sampler system as defined in the reference methods.

Summary of Corrective Actions for 6 Liter Cans Collected Using Flow Controllers

Can Pressure	Lab Action	PM Action	NCM Needed?	Narrative Content
If can received open	Notify PM by email.	1. Inform client of the potential contamination. 2. If cancelled, initiate NCM and email Air Group. 3. If not cancelled, email Air Group to proceed with analysis.	Yes, if analysis cancelled	1. If cancelled, narrate. 2. If analyzed, narrate possibility of contamination.
Ambient to -9.9 (200 mL) Ambient to -7.5 (500 mL)	Proceed with analysis.	None	No	None
-10 to -23.9 (200 mL) -7.6 to -23.9 (500 mL) (inches Hg)	1. Pressurize to above ambient. 2. Proceed with analysis; if possible, analyze more volume to lower the dilution factor.	None	No	If RL not achieved due to insufficient volume, narrate dilutions that were necessary due to insufficient sample.
-24 to -25 (inches Hg)	1. Pressurize to above ambient. 2. Verify flow controller was working properly. 3. Notify PM by email, wait for client response.	1. Notify client that can pressure should have been higher if flow control was used properly and that there is not enough sample for analysis without dilution. 2. If cancelled, initiate NCM and email Air Group. 3. If not cancelled, email Air Group to proceed with analysis.	Yes, if analysis cancelled	1. If cancelled, narrate 2. Narrate dilutions that were necessary due to insufficient sample.
-26 or lower (inches Hg)	1. If trip blank, pressurize to above ambient, proceed with analysis using DF = 1. 2. Verify flow controller was working properly. 3. If not a trip blank, pressurize to above ambient. Notify PM by email, wait for client response.	1. Notify client that can pressure should have been higher if flow controller was used properly and that there is not enough sample for analysis without dilution. 2. If cancelled, initiate NCM and email Air Group. 3. If not cancelled, email Air Group to proceed with analysis.	Yes, if analysis cancelled	1. If cancelled, narrate. 2. Narrate dilutions that were necessary due to insufficient sample, dilution factor/results are estimated. "EST" flag on all detects

Summary of Corrective Actions for 6 Liter Cans Collected NOT Using Flow Controllers ("grab" sampling)

Can Pressure	Lab Action	PM Action	NCM Needed?	Narrative Content
If can received open	Notify PM by email.	1. Recommend to client that sample be cancelled due to possible contamination. 2. If cancelled, initiate NCM and email Air Group. 3. If not cancelled, email Air Group to proceed with analysis.	Yes, if analysis cancelled	1. If cancelled, narrate 2. If analyzed, narrate possibility of contamination.
Ambient to -9.9 (200 mL) inches Hg Ambient to -7.5 (500 mL) inches Hg	Proceed with analysis.	None	No	None
-10 to -23.9 (200 mL) -7.6 to -23.9 (500 mL) (inches Hg)	1. Pressurize to above ambient. 2. Proceed with analysis; if possible analyze more volume to lower the dilution factor.	None	No	If RL not achieved due to insufficient volume, narrate dilutions that were necessary due to insufficient sample
-24 to -25 (inches Hg)	1. Pressurize to above ambient. 2. Notify PM by email, wait for client response.	1. Notify client that can pressure should have been higher if grab can was used properly and that there is not enough sample for analysis without dilution. 2. If cancelled, initiate NCM and email Air Group. 3. If not cancelled, email Air Group to proceed with analysis.	Yes, if analysis cancelled	1. If cancelled, narrate 2. Narrate dilutions that were necessary due to insufficient sample.
-26 or lower	1. If trip blank, pressurize to above ambient, proceed with analysis using DF = 1. 2. If not a trip blank, pressurize to above ambient. Notify PM by email, wait for client response.	1. Notify client that can pressure should have been higher if grab can was used properly and that there is not enough sample for analysis without dilution. 2. If cancelled, initiate NCM and email Air Group. 3. If not cancelled, email Air Group to proceed with analysis.	Yes, if analysis cancelled	1. If cancelled, narrate 2. Narrate dilutions that were necessary due to insufficient sample, dilution factor/results are estimated. "EST" flag on all detects

11.1.2. If the sample must be pressurized, attach the humidified nitrogen line to the can using a quick-connect fitting. Turn on the nitrogen, adjust the line pressure to approximate desired final pressure, and open the can. Remove the nitrogen line and connect a pressure gauge to obtain the pressure reading.

11.1.3. Measure the initial and final pressure/vacuum of the canisters using an NIST traceable, certified vacuum and/or pressure gauge.

11.1.4. The barometric pressure, initial pressure/vacuum and final pressure/vacuum are recorded in a laboratory worksheet and used to

calculate the dilution factor caused by pressurizing the can to working conditions.

11.1.5. The canister is allowed to equilibrate for approximately one hour before analysis. If the canister was pressurized to greater than 15 psig, pressure is released from the canister to bring the pressure below 15 psig. For autosampler volumes scheduled to be below 50 mL, the can pressure must be reduced to below 7 psig to more accurately measure the volume injected.

11.1.6. If necessary, this canister is further diluted by the dilution methods discussed in sections 11.3, 11.5, and 11.6.

11.2. Following a successful initial or calibration verification and prior to analysis of actual samples, an acceptable system blank and LCS must be analyzed (see sections 9.2 and 9.3). Following successful system blank and LCS analysis, actual sample analysis may begin. The LCS and blank are analyzed every 24 hour tune or every 20 samples, whichever is more frequent.

11.2.1. The desired sample size of each sample to be analyzed is determined by screening the cans according to SOP KNOX-MS-0010, current revision, Volatile Analyte Screening By Purge and Trap. The standard aliquot size is 200 mL for standard reporting limit work or 500 mL for low-level work. Sample volume injected can range from 10 mL to 1000 mL. For sample volumes below 50 mL, the can pressure must be reduced to below 7 psig to more accurately measure the volume injected. Volumes larger than 1000 mL can cause trap freeze-up when high humidity samples are trapped. If samples have been adequately pressurized with nitrogen, have been diluted, or only a small amount of sample collected in the can, then volumes larger than 1000 mL may be trapped, and the internal standards and surrogate monitored closely for breakthrough or freeze-up problems.

11.2.2. The pressure of each sample canister is checked. If the pressure is above 15 psig, the excess pressure is vented.

11.2.3. Each sample, volume (aliquot), method, and autosampler position are entered into the Entech sequence table.

11.2.4. If necessary, the automated flush function is used to sweep each autosampler line in the name list with helium.

11.2.5. The cans are then securely tightened onto the autosampler with the canister valves closed.

11.2.6. The sample work order numbers, and other pertinent information such as the GC method and the processing method are entered into the GC/MS sequence table.

11.2.7. The sample volume programmed, the can number, and the can's dilution factor are noted in the analytical run log. The Entech sequence, the GC/MS sequence table and the run log are verified to be in order.

11.2.7.1. If the actual sample amount trapped is greater than 5% from the programmed volume, the actual volume trapped is documented and used in calculating the results.

11.2.8. The Entech autosampler is started and the GC/MS acquisition program is started. (Note: The scan and GC parameters are controlled by the GC/MS method.)

11.2.9. 40 ml of the surrogate/internal standard is trapped on the Entech concentrator prior to sample introduction.

11.2.10. The analysis proceeds automatically for each name in the Entech autosampler program.

11.2.11. The internal standards and surrogate must pass all the criteria specified in section 9.1.

11.3. Autosampler Dilutions

11.3.1. Volumes of 10 to 1000 mL may be analyzed by the autosampler (see section 11.2.1). The standard aliquot is 200 mL for standard reporting limit work and 500 mL for low-level work.

11.3.2. If an analyte found in the sample is over the curve by less than a factor of twenty (based on 200 mL nominal volume) or fifty (based on 500 mL nominal volume), then the aliquot size of the sample may be reduced to a volume as low as 10 mL. This dilution factor is multiplied with all other dilution factors for this sample to obtain the final dilution factor.

11.3.3. If a dilution is performed to bring one or more analytes within the calibration range, the analyte having the highest concentration should not be diluted to less than 20% of the calibration range unless there are significant amounts of non-target compounds present.

11.3.4. If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non target peaks are less than five times the height of the internal standards, then the sample is

reanalyzed at a more concentrated dilution (up to the nominal volume). This requirement is approximate and subject to analyst judgment.

11.3.5. Only the most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

11.4. Water addition

11.4.1. Humidity plays an important role in the recovery of certain target compounds, particularly polar compounds, and water is added to canisters where appropriate. The addition of water helps to stabilize the behavior of these compounds, which might otherwise interact with the interior surface of the canister or with the stainless-steel lines of the sample manifold.

11.4.2. Since it is not practical to know the relative humidity of all canisters received at the laboratory, canisters are assumed to be approximately 80 percent relative humidity. When making canister dilutions (see Sections 11.5, and 11.6), the analyst attempts to preserve the relative humidity of canisters at a level that will minimize recovery loss due to low canister relative humidity.

11.4.3. Under normal laboratory conditions, a 6 liter canister at ambient pressure will have a relative humidity of 100 percent if approximately 100 uL of water is in the canister.

11.4.3.1. The minimum relative humidity at which canisters containing polar analytes can be analyzed before polar target recovery is negatively affected is approximately 20 - 30 percent.

11.4.3.2. The minimum relative humidity at which canisters containing non-polar analytes can be analyzed before non-polar target recovery is negatively affected is approximately 10 percent.

11.5. Serial Dilution

11.5.1. High-level samples, for example, are those containing ppm levels of volatile organic compounds.

11.5.2. The original sample canister must have a positive pressure. If the pressure is less than 0 psig, then proceed to Section 11.1.2.

11.5.3. A septum cap is attached to the sample canister and a gas-tight syringe is purged with UHP nitrogen. A septum cap is attached to a clean evacuated 6-liter canister (the dilution canister).

11.5.4. The syringe is inserted into the septum cap of the canister containing the sample and the canister valve is opened. The syringe is purged twice with sample and vented. The desired volume is then withdrawn and transferred into the dilution canister. The dilution canister is then pressurized using humidified nitrogen.

11.5.5. The final pressure is measured in the serial dilution canister using a NIST traceable, certified gauge.

11.5.6. If the canister was pressurized to greater than 15 psig, pressure is released from the canister to bring the pressure below 15 psig.

11.5.7. The barometric pressure, the aliquot volume, final canister pressure and canister serial number is recorded in a laboratory worksheet. The serial dilution factor is calculated.

11.5.8. If a high level dilution is performed to bring one or more analytes within the curve, the analyte having the highest concentration should not be diluted to less than 20% of the upper calibration range, unless there are significant amounts of non-target compounds present. It is imperative that high levels of target and non-target analytes not contaminate the analytical system.

11.5.9. This serial dilution canister may be further diluted, if necessary, by another serial dilution, in-can dilution (see section 11.6), or on the autosampler (see section 11.3). The final dilution factor is the product of all the dilution factors for the sample.

11.6. In-canister Dilutions

11.6.1. If an analyte found or suspected to be in the sample is over the calibration range, to a level that an autosampler dilution would be insufficient, an in-canister dilution may be performed.

11.6.2. The canister vacuum/pressure is checked. If the can is under vacuum, then record the vacuum reading and proceed to section 11.6.3. If the canister is under pressure, then the can is bled to ambient pressure, then proceed to section 11.6.3.

11.6.3. The canister is pressurized to the desired pressure with humidified nitrogen. The pressure must be no more than 40 psig.

11.6.4. The final pressure is measured using an NIST traceable, certified gauge.

11.6.5. The barometric pressure and the final pressures are recorded in a laboratory worksheet and the in-can dilution factor is calculated. (note: if multiple in-can dilutions are performed, record the final pressure of each pressurization before venting to perform the subsequent pressurization).

11.6.6. If the final pressure of the canister was greater than 15 psig, pressure is released from the canister to bring the pressure below 15 psig before loading on the autosampler.

11.6.7. If an in-canister dilution is performed to bring one or more analytes within the curve, the analyte having the highest concentration must not be diluted to less than 20% of the upper calibration range, unless there are significant amounts of non-target compounds present. Care must be taken to avoid over-dilution for in-canister dilutions since the original sample is affected.

11.6.8. This in-can dilution canister may be further diluted, if necessary, by another in-can dilution, or a serial dilution (see section 11.5), or on the autosampler (see section 11.3). This dilution factor is multiplied with all other dilution factors for this sample to obtain the final dilution factor.

11.7. Troubleshooting and Maintenance.

11.7.1. Troubleshooting is the identification and elimination of a problem or malfunction in a system or process. Troubleshooting is most effective when it is performed in a logical step by step sequence. (Refer to the instrument manufacturer's manual for specific guidance)

11.7.2. Basic Troubleshooting Principles

- Identify and analyze the problem and symptoms.
- Gather information that may help identify the problem such as prep information, system log, or output files.
- Evaluate the possibility that recent changes to the system caused the problem.
- Try to reproduce the problem if at all possible.
- Eliminate as many variables as possible.
- Document the system state then change variables one at a time and evaluate the effect on the problem

11.7.3. Major Maintenance

11.7.3.1. A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning or repairing the source, replacing filaments, changing electronics, replacing the multiplier or changing moisture or Tenax traps.

11.7.4. Minor Maintenance

11.7.4.1. Minor maintenance includes cleaning the injector port, replacing filters, changing the pump oil, autotuning, switching filaments (instrument contains two filaments under vacuum), replacing valves or rotors, change/refill the calibration vial, changing seals and o-rings, ballasting pump, replacing fuses, replacing roughing pumps, changing pump oil, or replacing transfer lines.

11.8. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure, except those specified by project specific instructions, shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist, Project Manager and QA Manager. If contractually required, the client shall be notified.

11.9. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.10. Refer to TestAmerica Knoxville SOP KNOX-IT-0001, current revision, for requirements for computer hardware and software.

12. Data Analysis and Calculations

12.1. Refer to Figure 2 for an example data review checklists used to perform and document the review of the data. Using the data review checklist, the analyst also creates a narrative which includes any qualifications of the sample data.

12.2. Tentatively Identified Compounds (TICs): Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) are performed if required by the client. They are evaluated using the TestAmerica Knoxville SOP KNOX-MS-0014, current revision, "Determination of Tentatively Identified Compounds (TICs)"

12.3. Qualitative Identification: An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the most recent NIST library.

Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion

due to co-elution is evaluated). The characteristic ions from the reference mass spectrum are defined as the three ions with greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions are present in the reference spectrum (i.e. characteristic ions have relative intensity >30%).

- The sample component retention time must compare to within +0.2 minute of the retention time of the standard component. For reference, the standard must be run within the same 24 hours as the sample.
- The characteristic ions of a compound must maximize in the same scan or within one scan of each other.
- The relative intensities of ions should agree to within +30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%).

  If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst reports that identification and proceeds with quantitation.

12.4. Calculation legend:

A	=	amount of neat compound, uL
CB	=	concentration in SGDB, ug/mL
CC	=	concentration in canister, ppb v/v
CS	=	concentration in mix, ug/uL
Cx	=	the value determined by vendor certification analyses is used in the following calculations, ppb v/v (1000 ppb nominal)
d	=	density of neat compound, g/mL
DF	=	dilution factor, unitless
FV	=	final volume in a pressurized canister, L
GC	=	gas constant at 25°C and standard pressure, 24.45 nL/n mole (or g/mol)
MW	=	molecular weight, ng/n mole
P_B	=	barometric pressure
P_F	=	final pressure, units specified
P_I	=	initial pressure, units specified
P_T	=	transfer pressure, units specified
Px	=	pressure in X = inches, psia or mmHg
TK	=	temperature in Kelvin
TV	=	transfer volume, L, mL or uL
Vbottle	=	volume of static gas dilution bottle, mL
Vmix	=	volume of mix, □L

Pmm Hg = P inches x 25.4
P inches = Ppsi * 2.036

Pmm Hg = P_{psi} x 51.7149

12.5. Calculations:

12.5.1. Final Canister Volume

$$FV = \frac{Canister\ size(L)\ x\ P_F(mm\ Hg\ Abs)}{P_B(mm\ Hg\ Abs)}$$

12.6. Stock standards in SGDB

12.6.1. Liquid formula

$$CB_{LIQUID}, \square g/mL = \frac{\#\square L * d * 1000ug/mg}{V_{bottle}}$$

12.6.2. Solid formula

$$CB_{SOLID}, \square g/mL = \frac{\#\ mg * 1000ug/mg}{V_{bottle}}$$

12.7. Concentration of standards in primary target standard made from SGDB

$$CC, ppb\ v/v = \frac{TV, mL * CB\ x\ 1000(g/ug)(nL/L) * GC}{FV * MW * P_F atm(seenote)}$$

note:Atm = ("Hg gauge + 28.735 "Hg) / 28.735 "Hg
where 28.735 = barometric pressure based on STP corrected for Knoxville elevation of 305 m
STP = 760 mm Hg
Subtract 30.1 for elevation (from "Reduction of Barometer to Sea Level" pg 15-13, *Handbook of Chemistry and Physics*)
= 729.87 mm Hg
= 28.735 "Hg

12.8. Concentration of Analytes in Primary Target Standard (gauge method)

$$CC, ppb\ v/v = \frac{(P_T - P_I, inchesHg)(Cx\ or\ CC)}{(P_F, inchesHg + P_B, inchesHg)}$$

12.9. Concentration of Analytes in Primary Target Standard (syringe method)

12.10. Concentration of Analytes in Primary Target Standard (mass flow controller

$$CC,\ ppb\ v/v = \frac{(TV, mL)(CC\ or\ Cx)}{(FV, mL)}$$

method)

12.10.1. The calculation is the same as 12.9 except to calculate *TV,mL*, through the mass flow controller:

TV,mL =flow rate (mL/min) * min

12.11. Dilution Factors of original sample canisters

12.11.1. In Can Dilution Factor

$$DF = \frac{P_{f(mm\,Abs)}}{P_{i(mm\,Abs)}}$$

12.11.2. Serial Dilution Factor

DF = FV/TV, (L)

12.11.3. Instrument Dilution Factor

$$\text{DF} = \frac{\text{Nominal Sample Volume}}{\text{Sample Volume Injected}}$$

12.12. Response Factor (RF)

$$RF = \frac{Ax * Cis}{Ais * Cx}$$

where:

x	=	area of the characteristic ion for the target compound.
Ais	=	area of the characteristic ion for the internal standard.
Cx	=	amount of the target compound.
Cis	=	amount of the internal standard.

12.13. Average Response Factor (ARF)

$$ARF = \frac{RF_1 + RF_2 + \ldots RF_n}{n}$$

where:
n = the number of calibration points

12.14. Standard deviation of the ARF:

$$S = \sqrt{\frac{\sum_i^n (ARF - RF_n)^2}{n-1}}$$

12.15. Calibration Curve Evaluation Calculations

12.15.1. Relative standard deviation (RSD) of the ARF:

$$RSD = \frac{S}{ARF} * 100\%$$

12.15.2. Coefficient of Determination (r^2)

$$r^2 = \frac{\sum_1^n (y_{obs} - \overline{y_{obs}})(y_{pred} - \overline{y_{pred}})}{\sqrt{\sum_1^n (y_{obs} - \overline{y_{obs}})^2 \sum_1^n (y_{pred} - \overline{y_{pred}})^2}}$$

where

y_{obs} = Concentration of initial calibration standard (for standards 1 through n)

$\overline{y_{obs}}$ = Average of concentrations of initial calibration standards

y_{pred} = Predicated concentration of initial calibration standard (for standards 1 through n)

$\overline{y_{pred}}$ = Average of predicated concentrations of initial calibration standards

(For y_{pred} refer to calculation of Cpv found in Sections 12.19.1.2 and 12.19.1.3)

12.16. Calibration Verification: Percent deviation (% D) of the daily RF values as compared with the initial ARF values:

$$\% D = \frac{|RF - ARF|}{ARF} * 100\%$$

12.17. Surrogate and Laboratory Control Sample percent recovery (%R):

$$\%R = \frac{FoundAmount,ppb}{SpikeAmount,ppb} * 100\%$$

12.18. Duplicate relative percent difference (RPD):

$$RPD = \frac{|A_1 - A_2|}{\overline{A}} x\ 100\%$$

where:

A_1 = amount determined in first analysis

A_2 = amount determined in second analysis

$\overline{A}$ = average determination, $(A_1 + A_2)/2$

12.19. Calibration verification percent drift and difference from the initial calibration:

$$\%\ Drift = \frac{C_{expected} - C_{found}}{C_{expected}} \square 100$$

Where

$C_{expected}$ □ Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

$$\%Difference \square \frac{\overline{RF} \square RF}{\overline{RF}} \square 100$$

$\overline{RF}$ □ Average Analyte Response Factor from Initial Calibration

RF = Measured Analyte Response Factor from Calibration Verification

12.19.1. Target analyte concentrations in samples are typically calculated using the average response factor from the initial calibration. Quantitation may also be determined using linear or second order curves at the analyst's discretion to improve the quantitation of target analytes.

12.19.1.1. Calculation of concentration using Average Response Factors

$$C_{pv} \square \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

12.19.1.2.Calculation of concentration using Linear fit

$$C_{pv} = A + B\frac{R_x C_{is}}{R_{is}}$$

C_{pv} = Concentration, ppb (v/v)
R_x = Response for analyte (area of quantitation ion)
R_{is} = Response for internal standard (area of quantitation ion)
C_{is} = Concentration of internal standard
A = Intercept
B = Slope

The corresponding Target software calculation is as follows:

$$Cpv = Cis\left(b + \frac{1}{m1} * \frac{Rx}{Ris}\right)$$

b = Concentration Ratio Intercept
m1 = Inverse of Slope

12.19.1.3.Calculation of concentration using Quadratic fit

$$Cpv = A + B\left(\frac{RxCis}{Ris}\right) + C\left(\frac{RxCis}{Ris}\right)^2$$

C = Curvature

The corresponding Target software calculation is as follows:

$$Cpv = Cis\left(b + m1 * \frac{Rx}{Ris} + m2 * \left(\frac{Rx}{Ris}\right)^2\right)$$

m1 = First order coefficient
m2 = Curvature (Second order coefficient)

12.20. Sample Quantitation: The amount of target compound detected is determined using the average RF or calibration curve values from the initial calibration (not the continuing calibration):

$$\text{Amount} = Cpv * DF$$

12.21. Unit conversions

12.21.1.

$$\text{Amount}, \mu g/m^3 = \frac{\text{Amount, ppb (v/v)} * MW}{GC}$$

12.21.2.

$$\text{Amount, ppm v/v} = \text{amount,} \frac{\text{ppb (v/v)}}{1000}$$

13. Method Performance

13.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte in each routine matrix prior to the analysis of any samples. The procedure for determination of the method detection limit is given in the SOP CA-Q-S-006 current revision based on 40 CFR Part 136 Appendix B. The result of the MDL determination must support the reporting limit. MDL summaries are stored on the local area network.

13.2. Initial Demonstration of Capability – Each analyst must perform an initial demonstration of capability (IDOC) for each target analyte prior to performing the analysis independently. The IDOC is determined by analyzing four replicate spikes (e.g., LCSs) as detailed in TestAmerica Knoxville SOP KNOX-QA-0009. Recovery limits must be 70-130% and RSD must be less than or equal to 25%. Recovery limits for butane and propene are 60-140% and RSD must be less than or equal to 30%.

13.3. Training Qualification: The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. Refer to SOP KNOX-QA-0009 current revision for further requirements for performing and documenting initial and on-going demonstrations of capability.

14. Pollution Prevention

14.1. All attempts will be made by laboratory personnel to minimize the use of solvents when performing this procedure.

15. Waste Management

15.1. All waste will be disposed of in accordance with all Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. The following waste streams are produced when this method is carried out.

- Expired solid and liquid standards are stored in metal closed-top containers.

16. References

16.1. Compendium Method TO-14, "The Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using SUMMA® Passivated Canister Sampler and Gas Chromatographic Analysis," U.S. EPA 600/4-89/017, June 1988.

16.2. Compendium Method TO-14A, "Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using Specially Prepared Canisters With Subsequent Analysis by Gas Chromatography," U.S. EPA 625/R-96/010b, January 1999.

16.3. Compendium Method TO-15, "Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GCMS)", U.S. EPA 625/R-96/010b, January 1999.

16.4. TestAmerica Quality Assurance Manual (QAM), current revision.

16.5. Entech Instruments Inc. 7100 Operators Manual. Version 2.0 for the 7100 Preconcentrator and Accessories

16.6. Agilent HP 5973 and 6890 Operation Manuals for GC and GC/MS.

17. Miscellaneous

17.1. Other SOPs cross-referenced in this SOP:

- KNOX-MS-0022, "Canister Cleaning and Preparation," latest revision.
- KNOX-MS-0010, "Volatile Analyte Screening by Purge and Trap," latest revision.

17.2. Modification from the referenced methods

17.2.1. The TO-14A tune limits are more stringent than the TO-15 limits. The default procedure is to use the TO-14A limits for both TO-14A and TO-15 samples. However if all the samples analyzed in the 24-hour tune batch are TO-15 samples, the analyst may elect to use the TO-15 limits. This SOP also allows for 50 ng or less of BFB to verify tuning of the instrument.

17.2.2. The continuing calibration listed in this procedure allows target analytes over 30% D as long as the target analytes meet the LCS criteria, with a narrative note of those target analytes that are over 30% D.

17.2.3. This procedure uses purified nitrogen in place of zero humid air specified in the reference methods. This must be noted in the case narrative.

17.2.4. TO-14 requires that the RT shift for the internal standards at each calibration level must be within 20 seconds of the RT of the mid-level calibration for each internal standard. TO-15 specifies that the comparison is made to the mean RT over the initial calibration range for each internal standard. This SOP uses the TO-15 criteria.

17.2.5. Section 7.13 Method TO-15 states that the working standard may be stored for 30 days. This laboratory experience has allowed the standard expiration date to be 2 months with no significant degradation of the standards.

17.2.6. Surrogates are not required by the reference methods. This SOP adds surrogate bromofluorobenzene (BFB) to every sample to help monitor for matrix effects and method performance.

17.2.7. The TO-15 method states that the scan time must give 10 scans per peak, not to exceed 1 second per scan. The GC/MS software is set for a sampling rate of 3, which corresponds to approximately 2 to 3 scans per second, depending on the instrument. See the GC/MS operator's manual or "help" on the software for more information about the sampling rate.

17.2.8. EPA Method TO-14A specifies that the relative accuracy of the field sampler or sample delivery system must meet 90-110% for a standard at 8 ppb v/v. The laboratory Control Sample (LCS) summary data is evaluated against alternate acceptance criteria based on this laboratory procedure for method TO-14A. When TO-14A work is performed, this must be noted in the case narrative.

17.2.9. The TO-15 method states that for the internal standard (IS) of the method blank, The retention time for each of the IS must be within ±0.33 minutes between the blank and the most recent valid calibration, and the area response for each IS in the blank must be within ±40 percent of the mean area response of the IS in the most recent valid calibration. This SOP states the method blank IS are compared to the most recent continuing calibration standard.

17.3. List of Appendices

17.3.1. Appendix I: Target Analyte Tables

17.3.1.1. Table 1: Target Analytes - TO-14 and TO-15 Compounds

17.3.2. Appendix II: Figures

17.3.2.1.Figure 1: BFB Tuning Criteria

17.3.2.2.Figure 2: Example of a Data Review Checklist

17.3.2.3.Figure 3: Flow Chart

17.3.3. Appendix III: Example Instrument Parameters

17.3.4. Appendix IV: Recommended Calibration Levels (200 mL sample volume)

Appendix I: Table 1: Target Analytes

CAS NUMBER	TestAmerica Knoxville Compounds	MOLECULAR WEIGHT (ng/nmole)	conversion factor for ug/m3 = MW/24.45	200 mL REPORT-ING LIMIT (ppb(v/v)	200 mL REPORT-ING LIMIT (ug/m3)	500 mL REPORT-ING LIMIT (ppb(v/v)	500 mL REPORT-ING LIMIT (ug/m3)	SUGGESTED ION
71-55-6	1,1,1-Trichloroethane	133.4	5.45603	0.20	1.1	0.080	0.44	97
79-34-5	1,1,2,2-Tetrachloroethane	167.85	6.86503	0.20	1.4	0.080	0.55	83
79-00-5	1,1,2-Trichloroethane	133.4	5.45603	0.20	1.1	0.080	0.44	97
76-13-1	1,1,2-Trichlorotrifluoroethane [e]	187.37	7.66339	0.20	1.5	0.080	0.61	101
75-34-3	1,1-Dichloroethane	98.96	4.04744	0.20	0.81	0.080	0.32	63
75-35-4	1,1-Dichloroethene	96.94	3.96483	0.20	0.79	0.080	0.32	96
87-61-6	1,2,3-Trichlorobenzene[l]	181.45	7.42127	1.0	7.4	0.40	3.0	180
96-18-4	1,2,3-Trichloropropane[l]	147.43	6.02986	0.50	3	0.20	1.2	110
120-82-1	1,2,4-Trichlorobenzene[l]	181.45	7.42127	1.0	7.4	0.40	3.0	180
95-63-6	1,2,4-Trimethylbenzene	120.19	4.91575	0.20	0.98	0.080	0.39	105
106-93-4	1,2-Dibromoethane (EDB)	187.86	7.68344	0.20	1.5	0.080	0.61	107
95-50-1	1,2-Dichlorobenzene	147	6.01227	0.20	1.2	0.080	0.48	146
107-06-2	1,2-Dichloroethane	98.96	4.04744	0.20	0.81	0.080	0.32	62
78-87-5	1,2-Dichloropropane	112.99	4.62127	0.20	0.92	0.080	0.37	63
76-14-2	1,2-Dichlorotetrafluoroethane [c,i]	170.92	6.99059	0.20	1.4	0.080	0.56	135
108-67-8	1,3,5-Trimethylbenzene	120.19	4.91575	0.20	0.98	0.080	0.39	120
106-99-0	1,3-Butadiene[l]	54.09	2.21227	0.40	0.88	0.16 0.35		54
541-73-1	1,3-Dichlorobenzene	147	6.01227	0.20	1.2	0.080	0.48	146
106-46-7	1,4-Dichlorobenzene	147	6.01227	0.20	1.2	0.080	0.48	146
123-91-1	1,4-dioxane[l]	88.11	3.60368	0.50	1.8	0.20	0.72	88
71-36-3	1-Butanol[l]	74.12	3.03149	2.0	6.1	0.80	2.4	31
540-84-1	2,2,4-Trimethylpentane	114.23	4.67198	0.50	2.3	0.20	0.93	57
78-93-3	2-Butanone[l]	72.11	2.94928	1.0	2.9	0.40	1.2	72
95-49-8	2-chlorotoluene	126.58	5.17710	0.40	2.1	0.16	0.83	126
591-78-6	2-Hexanone[l]	100.16	4.09652	0.50	2.0	0.20	0.82	58
78-78-4	2-Methyl butane	72.15	2.95092	0.50	1.5	0.20	0.59	43

CAS NUMBER	TestAmerica Knoxville Compounds	MOLECULAR WEIGHT (ng/nmole)	conversion factor for ug/m3 = MW/24.45	200 mL REPORT-ING LIMIT (ppb(v/v)	200 mL REPORT-ING LIMIT (ug/m3)	500 mL REPORT-ING LIMIT (ppb(v/v)	500 mL REPORT-ING LIMIT (ug/m3)	SUGGESTED ION
107-05-1	3-Chloropropene[l]	76.52	3.12965	0.20	0.63	0.080	0.25	39
622-96-8	4-ethyltoluene	120.19	4.91575	0.40	2.0	0.16	0.79	105
108-10-1	4-Methyl-2-Pentanone[l]	100.16	4.09652	0.50	2.0	0.20	0.82	43
67-64-1	Acetone[l]	58.08	2.37546	5.0	12	2.0	4.8	58
75-05-8	Acetonitrile[l]	41.05	1.67894	1.0	1.7	0.40	0.67	40
107-02-8	Acrolein[l]	56.06	2.29284	1.0	2.3	0.40	0.92	56
107-13-1	Acrylonitrile[l]	53.06	2.17014	2.0	4.3	0.80	1.7	53
98-83-9	alpha-Methylstyrene[l]	118.18	4.83354	0.40	1.9	0.16	0.77	118
71-43-2	Benzene	78.11	3.19468	0.20	0.64	0.080	0.26	78
100-44-7	Benzyl Chloride	126.58	5.17710	0.40	2.1	0.16	0.83	91
75-27-4	Bromodichloromethane	163.83	6.70061	0.20	1.3	0.080	0.54	83
75-25-2	Bromoform[l]	252.73	10.3366	0.20	2.1	0.080	0.83	173
74-83-9	Bromomethane	94.94	3.88303	0.20	0.78	0.080	0.31	94
75-15-0	Carbon Disulfide	76.14	3.11411	0.50	1.6	0.20	0.62	76
56-23-5	Carbon Tetrachloride	153.82	6.29121	0.20	1.3	0.080	0.50	117
108-90-7	Chlorobenzene	112.56	4.60368	0.20	0.92	0.080	0.37	112
75-45-6	Chlorodifluoromethane [a,l]	86.47	3.53661	0.20	0.71	0.080	0.28	67
75-00-3	Chloroethane	64.51	2.63845	0.20	0.53	0.080	0.21	64
67-66-3	Chloroform	119.38	4.88262	0.20	0.98	0.080	0.39	83
74-87-3	Chloromethane[l]	50.49	2.06503	0.50	1.0	0.20	0.41	52
156-59-2	cis-1,2-Dichloroethene	96.94	3.96483	0.20	0.79	0.080	0.32	96
10061-01-5	cis-1,3-Dichloropropene	110.97	4.53865	0.20	0.91	0.080	0.36	75
98-82-8	Cumene [l]	120.19	4.91575	0.40	2.0	0.16	0.79	105
110-82-7	Cyclohexane	84.16	3.44213	0.50	1.7	0.20	0.69	69
124-18-5	Decane[l]	142.28	5.81922	1.0	5.8	0.40	2.3	57
124-48-1	Dibromochloromethane	208.28	8.51861	0.20	1.7	0.080	0.68	129
74-95-3	Dibromomethane	173.83	7.10961	0.40	2.8	0.16	1.1	93
75-71-8	Dichlorodifluoromethane[b,l]	120.91	4.94519	0.20	0.99	0.080	0.40	85

CAS NUMBER	TestAmerica Knoxville Compounds	MOLECULAR WEIGHT (ng/nmole)	conversion factor for ug/m3 = MW/24.45	200 mL REPORT-ING LIMIT (ppb(v/v)	200 mL REPORT-ING LIMIT (ug/m3)	500 mL REPORT-ING LIMIT (ppb(v/v)	500 mL REPORT-ING LIMIT (ug/m3)	SUGGESTED ION
141-78-6	Ethyl acetate[l]	88.11	3.60368	2.0	7.2	0.80	2.9	43
60-29-7	Ethyl Ether[l]	74.12	3.03149	2.0	6.1	0.80	2.4	31
100-41-4	Ethylbenzene	106.17	4.34233	0.20	0.87	0.080	0.35	91
87-68-3	Hexachlorobutadiene[l]	260.76	10.6650	1.0	11	0.40	4.3	225
67-63-0	Isopropyl alcohol[l]	60.1	2.45808	2.0	4.9	0.80	2.0	45
136777-61-2	m/p-Xylene [g, h]	106.17	4.34233	0.20	0.87	0.080	0.35	91
80-62-6	Methyl methacrylate[l]	100.12	4.09489	0.50	2.0	0.20	0.82	41
75-09-2	Methylene Chloride [f]	84.93	3.47362	0.50	1.7	0.20	0.69	84
1634-04-4	Methyl-tert-Butyl ether[l]	88.15	3.60532	1.0	3.6	0.40	1.4	73
91-20-3	Naphthalene[l]	128.17	5.24213	0.50	2.6	0.20	1.0	128
106-97-8	n-Butane[l]	58.12	2.37710	0.40	0.95	0.16 0.38		43
104-51-8	n-Butylbenzene[l]	134.22	5.48957	0.40	2.2	0.16	0.88	91
112-40-3	n-Dodecane[l]	170.33	6.96646	1.0	7.0	0.40	2.8	57
142-82-5	n-Heptane	100.2	4.09816	0.50	2.1	0.20	0.82	71
110-54-3	n-Hexane (f)	86.18	3.52474	0.50	1.8	0.20	0.70	56
111-65-9	n-Octane	114.23	4.67198	0.40	1.9	0.16	0.75	85
111-84-2	Nonane[l]	128.26	5.24581	0.50	2.6	0.20	1.0	57
103-65-1	n-Propylbenzene	120.19	4.91575	0.40	2.0	0.16	0.79	120
1120-21-4	n-Undecane[l]	156.31	6.39305	1.0	6.4	0.40	2.6	57
95-47-6	o-Xylene [h]	106.17	4.34233	0.20	0.87	0.080	0.35	91
99-87-6	p-Cymene [k]	134.22	5.48957	0.20	1.1	0.08	0.44	119
109-66-0	Pentane	72.15	2.95092	1.0	3.0	0.40	1.2	72
115-07-1	Propene[l]	42.08	1.72106	0.50	0.86	0.20 0.34		41
135-98-8	sec-butylbenzene	134.22	5.48957	0.40	2.2	0.16	0.88	105
100-42-5	Styrene	104.15	4.25971	0.20	0.85	0.080	0.34	104
75-65-0	Tert-Butanol[l]	74.12	3.03149	2.0	6.1	0.80	2.4	59
98-06-6	tert-butylbenzene	134.22	5.48957	0.50	2.7	0.20	1.1	119
127-18-4	Tetrachloroethene	165.83	6.78241	0.20	1.4	0.080	0.54	129

CAS NUMBER	TestAmerica Knoxville Compounds	MOLECULAR WEIGHT (ng/nmole)	conversion factor for ug/m3 = MW/24.45	200 mL REPORT-ING LIMIT (ppb(v/v)	200 mL REPORT-ING LIMIT (ug/m3)	500 mL REPORT-ING LIMIT (ppb(v/v)	500 mL REPORT-ING LIMIT (ug/m3)	SUGGESTED ION
109-99-9	Tetrahydrofuran[l]	72.11	2.94928	1.0	2.9	0.40	1.2	42
108-88-3	Toluene	92.14	3.76851	0.20	0.75	0.080	0.30	91
1330-20-7	Total-Xylenes	106.17	4.34233	0.40	1.7	0.16	0.70	91
156-60-5	trans-1,2-Dichloroethene	96.94	3.96483	0.20	0.79	0.080	0.32	96
10061-02-6	trans-1,3-Dichloropropene	110.97	4.53865	0.20	0.91	0.080	0.36	75
79-01-6	Trichloroethene	131.39	5.37382	0.20	1.1	0.040	0.22	130
75-69-4	Trichlorofluoromethane [d,l]	137.37	5.61840	0.20	1.1	0.080	0.45	101
108-05-4	Vinyl Acetate[l]	86.09	3.52106	1.0	3.5	0.40	1.4	43
593-60-2	Vinyl Bromide [j,l]	106.95	4.37423	0.20	0.87	0.080	0.35	106
75-01-4	Vinyl Chloride	62.5	2.55624	0.20	0.51	0.080	0.20	62

a) Freon 22
b) Freon 12
c) Freon 114
d) Freon 11
e) Freon 113, also 1,1,2-Trichloro-1,2,2-trifluoroethane
f) This is a common laboratory solvent
g) m-xylene and p-xylene coelute
h) Total xylenes (CAS # 1330-20-7) is the sum of m/p-xylenes and o-xylene
i) isopropylbenzene
j) bromoethene
k) isopropyltoluene
l) identified as provisory analyte. See Section 9.3.2.

Appendix II: Figures

Figure 1a: TO-14A BFB Tuning Criteria

Mass	Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base peak, 100% relative abundance
96	5 to 9% of mass 95
173	Less than 2% of mass 174
174	Greater than 50% of mass 95
175	5 to 9% of mass 174
176	Greater than 95% but less than 101% of mass 174
177	5 to 9 % of mass 176

Note: All ion abundances must be normalized to m/z 95, the nominal base peak, even though m/z 174 may be over 100 % of m/z 95.

Figure 1b: TO-15 BFB Tuning Criteria

Mass	Abundance Criteria
50	8.0 to 40.0 % of mass 95
75	30.0 to 66.0 % of mass 95
95	Base peak, 100% relative abundance
96	5.0 to 9.0 % of mass 95
173	Less than 2.0 % of mass 174
174	50.0 to 120.0 % of mass 95
175	4.0 to 9.0 % of mass 174
176	93.0 to 101.0 % of mass 174
177	5.0 to 9.0 % of mass 176

Note: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

Figure 2: Example Data Review Checklist

TestAmerica Knoxville GC/MS Air Initial Calibration Data Review / Narrative Checklist
Method: TO-14 and TO-15 - KNOX-MS-0001, Rev 14 & KNOX-MS-0023, Rev 1

Analysis Date:		Instrument::		ICAL Batch/Scan Name:		Scanned ¤

Review Items	N/A	Yes	No	If No, why is data reportable?	2nd
1. Did BFB meet tune criteria?					
2. Were all standards injected within 24 hr of BFB?					
3. Was date/time of analysis verified and logbook as correct?					
4. Is low level std at or <RL and are the remaining points consecutive?					
5. Are the calibration levels correct? (Calculate standard concentration & amt. injected with quan rpt at each level)					
6. Was ICAL processed using correct methods and files?					
7. Are the ICAL start and end dates/times correct?					
8. Were at least 5 levels of each compound analyzed?					
9. At least 6 consecutive points used for quadratic curves, and at least 5 consecutive points for linear curves? Note: Ohio does not allow Quad					
10. Is %RSD for all target analytes < 30%? (with up to 2 compounds with RSD < 40%)					
11. If curves were used, is correlation coefficient ≥0.990?					
12. For quadratic: is a tangent's slope to the curve entirely positive or negative and continuous					
13. For linear or quadratic: origin NOT "included"? (NOTE: OHIO does NOT allow "FORCE" through origin).					
14. Is the "Y" intercept less than the RL for each curve?					
15. RT for each IS ±20 sec avg. RT?					
16. Area for each IS ± 40% avg. area?					
17. Each analyte ± 0.06 RRT of avg. RRT?					
18. Have all peaks been auto identified? If not, list:					
19. If manual integrations were performed, are they clearly identified, initialed, dated and reason given?				Reasons: 1)Corrected split peak; 2)Unresolved peak; 3)tailing; 4)RT shift; 5)wrong peak selected; 6)other	
20. Have alternate hits/manual integrations been verified as correct and are correct RFs listed in ICAL summary?					
21. Are all the active compounds listed on each quan report?					
22. High point checked for saturation and point removed if saturated?					
23. Elution order checked on isomeric pairs?					
• dichlorodifluoromethane / 1,2-dichlorotetrafluoroethane					
• trichlorofluoromethane / 1,1,2-trichlorotrifluoroethane					
• vinyl acetate / hexane					
• cis- and trans- isomers					
• ethyl benzene / m/p-xylene / o-xylene					
• n-propylbenzene/4-ethyl toluene/1,3,5-trimethylbenzene/1,2,4-trimethylbenzene					
• tert-butylbenzene/p-cymene					
• 1,2,4-trimethylbenzene/sec-butylbenzene					
• 1,3- , 1,4- , and 1,2-dichlorobenzene					
• 1,2,4-trichlorobenzene/1,2,3-trichlorobenzene					
24. Is the second source analysis of a reference standard within limits? (65-135% R)					
25. If criteria were not met, was a NCM generated, approved by supervisor, and copy included in folder?					
26. Does the ICAL folder contain complete data in the following order: Data review checklist, a complete runlog, BFB info, ICAL summary, curves, followed by [Quan reports, chromatograms, manual integrations], in increasing amount order, 2nd source info.					

Analyst:	Date:	2nd Level Reviewer :	Date:
Comments:		Comments:	

Figure 2: Example Data Review Checklist (continued)

TestAmerica Knoxville GC/MS Air Continuing Calibration Review / Narrative Checklist
Method: TO-14 and TO-15 - KNOX-MS-0001, Rev 14 & KNOX-MS-0023, Rev 1

Analysis Date:		CCAL Batch/ Scan Name:		Instrument:		ICAL Batch/ Scan Name:		Scanned □

Review Items	N/A	Yes	No	If No, why is data reportable?	2nd
1. Did BFB meet tune criteria?				□ failed for TO-14A, but passes for TO-15	
2. Were all standards injected within 24 hr of BFB?					
3. Have the Entech position no. & vol. been verified with run log & sample vol. corrected if actual amount differs >5%?					
4. Was date/time of analysis in logbook correct?					
5. Was the CCAL compared to the correct ICAL (date & time on CCAL matches the ICAL)					
6. Is the %D ≤ 30% for all target analytes? (Narrative req'd.)				□ [ccal] analytes > 30% but passes LCS criteria.	
7. Have all peaks been auto identified? If not, list:					
8. If manual integrations were performed, are they clearly identified, initialed, dated and reason given?				Reasons: 1)Corrected split peak; 2)Unresolved peak; 3)tailing; 4)RT shift; 5)wrong peak selected; 6)other	
9. Have alternate hits/manual integrations been verified as correct and are correct RFs listed in CCAL summary?					
10. Is the first IS documented correctly on the log?					
11. Elution order checked on isomeric pairs?					
• dichlorodifluoromethane / 1,2-dichlorotetrafluoroethane					
• trichlorofluoromethane / 1,1,2-trichlorotrifluoroethane					
• vinyl acetate / hexane					
• cis- and trans- isomers					
• ethyl benzene / m/p-xylene / o-xylene					
• n-propylbenzene/4-ethyl toluene/1,3,5-trimethylbenzene/1,2,4-trimethylbenzene					
• tert-butylbenzene/p-cymene					
• 1,2,4-trimethylbenzene/sec-butylbenzene					
• 1,3- , 1,4- , and 1,2-dichlorobenzene					
• 1,2,4-trichlorobenzene/1,2,3-trichlorobenzene					
12. Did the LCS meet criteria (70-130% with a limited # allowed 60-140% (see table) provisional analyte limit 60-140% with a limited # allowed 50-150%, and no two consecutive MEs). Note: Ohio does not allow for ME				□ [lcs6] LCS analyte(s) flagged as being outside control limits but within marginal limits □ [lcs5] LCS outside marginal exceedences high, but analytes were not detected	
13. If criteria were not met, was a NCM generated, approved by supervisor, and copy included in folder?					
14. Does the CCAL folder contain **complete** data in the following order: data review checklist, a **complete** runlog, tune pass/fail page, m/z list, tune chromatogram, Target CCAL summary, Quan report, chromatogram, manual integrations.					

Number of target analytes in LCS	# marginal exceedances of LCS control limits allowed
≥90	5
71 - 90	4
51 - 70	3
31 - 50	2
11 - 30	1
≤ 10	0

Analyst:	Date:	2nd Level Reviewer :	Date:
Comments:		Comments:	

MS017r32, 062113

Figure 2: Example Data Review Checklist (continued)

TestAmerica Knoxville GC/MS Air Data Review / Narrative Checklist LOT/Project #_____
Method: TO-14 and TO-15 - KNOX-MS-0001, Rev 14 & KNOX-MS-0023, Rev 1

Instrument:				
Scanned File:				

Review Items **A. Tune / Continuing Calibration**	N/A	Yes	No	Why is data reportable?	2nd ☐
1. Were all samples injected within 24 hr of BFB?					
2. Has a Continuing Calibration Checklist & run log been completed for each analytical batch **and scanned properly**?					
3. Was the correct ICAL used for quantitation?					
B. CLIENT SAMPLE AND QC SAMPLE Results	**N/A**	**Yes**	**No**	**Why is data reportable?**	
1. Were all special project requirements met?					
2. Were samples received in cans?				☐ [Tedlar1] analyzed w/n 72 hours, ☐ [Tedlar2] X-fer within 72 hours.	
3. Can pressure/vac on receipt acceptable?				☐ see narrative	
4. Were dilution factors/can prep information verified?					
5. Have the can number & lab ID been verified between the analysis log & sample prep log?					
6. Sample analyses done within analytical holding time (HT)? *If no, list samples:* _____				☐ [ht2] Client requested analysis after HT expired. ☐ Other: _____	
7. Default sample volume verified?					
8. Are surrogates and internal standards within QC limits? (60-140% R for surr.; 60-140%R from CCAL for IS) *If no, list samples/reason (e.g.,* sur1*):* Sample Reason Sample Reason _____ _____ _____ _____ _____ _____ _____ _____				☐ [sur7] Obvious matrix effect ☐ **[sur12] high recovery, no hits.** ☐ [sur14] entire sample consumed ☐ **[is1] Per client, reanalysis was not performed *** ☐ [is2] Reanalysis confirmed a matrix effect.	
9. Were all positive results and false negatives on quan report verified to be correct in LIMS?					
10. For dilutions, is highest concentration hit ≥ 20% cal range and not above calibration range? *List samples and reason (e.g.,* elev1*):* Sample Reason Sample Reason _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____				☐ [elev1] Elevated RL for due to sample matrix interferences. ☐ [elev3] Elevated RLs for all analytes due to difficult sample matrix. ☐ [elev4] Elevated RLs based on screening ☐ [elev5] Elevated RLs for all analytes due to presence of non-target compounds. ☐ [elev7] Elevated RLs due to sample volume	
11. If manual integrations were performed, are they clearly identified, initialed, dated and reason given & alternate hits verified.				Reasons: 1)Corrected split peak; 2)Unresolved peak; 3)tailing; 4)RT shift; 5)wrong peak selected; 6)other	
C. Preparation QC					
1. System blank run every 24 hours prior to samples?					
2. System blank surrogate recoveries within QC limits (60-140% R) ?				☐ **[mb1] All sample surrogates OK and there is no analyte >RL in samples associated with blank.***	
3. Are all analytes present in the system blank < RL? (1/2 RL for DoD). *If no, list blank ID:* _____				☐ **[mb3] No analyte > RL in associated samples.*** ☐ **[mb4] Sample results > 10x higher than blank.**	
4. DUP done per 20 samples and are all RPDs within limits? (for target analytes >5x RL, <25% RPD; no criteria for methanol and n-butanol) *If no, list DUP ID:*					
5. Did the LCS meet criteria (70-130% with a limited # allowed 60-140% (see table) provisional analyte limit 60-140% with a limited # allowed 50-150%, and no two consecutive MEs). Note: Ohio does not allow for ME.				☐ [lcs6] LCS analyte(s) flagged as being outside control limits but within marginal limits ☐ **[lcs5] LCS outside marginal exceedences high, but analytes were not detected** LCS ID: _____	

Number of target analytes in LCS	# marginal exceedances of LCS control limits allowed
>90	5
71 - 90	4
51 - 70	3
31 - 50	2
11 - 30	1
< 11	0

D. Other			
1. Final report acceptable? (Results correct, RLs calculated correctly, units correct, surrogate %R correct, appropriate flags used, dilution factor correct, analysis dates correct.)			
2. Are all nonconformances documented appropriately and copy included with deliverable?			
4. Was a narrative prepared and all deviations noted?			☐ [1pt6]; ☐ [1pt11]; ☐ [1ptsur] ☐ [Extras]
5. TO14A Autotext included in narrative (for TO14A samples only).			☐ [TO14]
6. All target analytes on c.cal >30%D but passes LCS criteria noted in the narrative?			☐ [ccal] The ccal exhibited a %D ICAL >30% but passes LCS…list analytes on narrative.

Analyst:	**Date:**	**2nd Level Reviewer:**	**Date:**

☐ see following page for comments.

*Such action must be taken in consultation with client.

TestAmerica Knoxville GC/MS Air Data Review / Narrative Checklist LOT/Project #_______________
Method: TO-14 and TO-15 - KNOX-MS-0001, Rev 14 & KNOX-MS-0023, Rev 1

Analyst/date:	2nd level reviewer/date:

TestAmerica Knoxville GC/MS Air Data Review / Narrative Checklist LOT/Project #_____
Canister Blank Data
Method: TO-14 and TO-15 - KNOX-MS-0001, Rev 14 & KNOX-MS-0023, Rev 1

Instrument:				
Scanned File:				

Review Items **A. Tune / Continuing Calibration**	N/A	Yes	No	Why is data reportable?	2nd ☐
4. Were all blank checks injected within 24 hr of BFB?					
5. Has a Continuing Calibration Checklist & run log been completed for each analytical batch **and scanned properly**?					
6. Was the correct ICAL used for quantitation?					
B. CLIENT SAMPLE AND QC SAMPLE Results	**N/A**	**Yes**	**No**	**Why is data reportable?**	
1. Were all special project requirements met?					
2. Default sample volume verified?					
3. Were all positive results and false negatives on quan report verified to be correct in XL?					
4. Are all analytes present in the blank checks < RL? (1/2 RL for DoD). If no, list ID:_____				☐ [mb3] No analyte > RL in associated samples. ☐ [mb4] Sample results > 10x higher than blank.	
C. Preparation QC					
1. All target analytes on c.cal < 30%D? LCS criteria (70-130% with a limited # allowed 60-140% (see table) provisional analyte limit 60-140% with a limited # allowed 50-150%, and no two consecutive MEs). Note: Ohio does not allow for ME.				☐ [ccal] The ccal exhibited a %D >30% but passes LCS criteria. ☐ [ccal2] The ccal exhibited a %D >30% but all %D were high and target analytes were ND.	
D. Other					
1. Final report correct? (***Confirm can batch # & can ID***. Results correct, RL's calculated correctly, units correct, appropriate flags used.)					
2. Are all nonconformances documented appropriately and copy included with deliverable?					

Number of target analytes in LCS	# marginal exceedances of LCS control limits allowed
>90	5
71 - 90	4
51 - 70	3
31 - 50	2
11 - 30	1

Analyst/date:	2nd level reviewer/date:

*Such action must be taken in consultation with client.

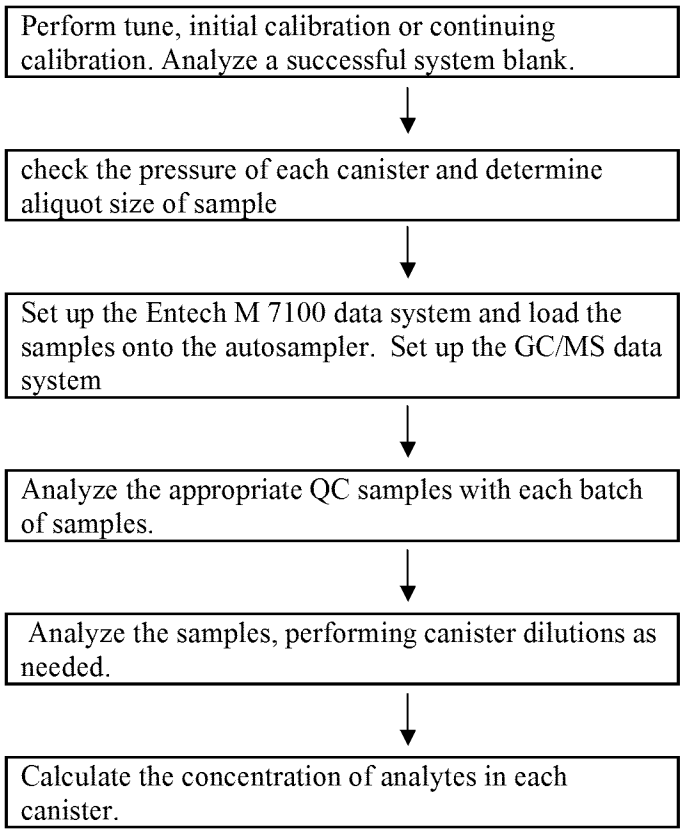
Figure 2: Example Data Review Checklist (continued)

TestAmerica Knoxville GC/MS Air Data Review
Additional ICAL Review Items (Internal Review)
Method: TO-14 and TO-15 - KNOX-MS-0001, Rev 14 & KNOX-MS-0023, Rev 1

TestAmerica Knoxville
New York Standard Low Reporting Limit Compounds
TO-15/TO-14A 500 ml analysis

Compound	RL(ppb/v)	1st Level	2nd Level
2-Butanone (MEK)	0.32		
tert-Butyl alcohol	0.32		
Carbon tetrachloride	0.040		
Hexachlorobutadiene	0.080		
Methyl tert-butyl ether	0.16		
1,2,4-Trichlorobenzene	0.080		
acrolein	0.16		
PCE	0.04		
TCE	0.04		
VC	0.04		

Figure 3: Flow Chart



Appendix III: Example Instrument parameters

```
                              TOPLEVEL PARAMETERS
                              -------------------

Method Information For: C:\MSDCHEM\1\METHODS\TO14.M

Method Sections To Run:

   ( ) Save Copy of Method With Data
   ( ) Pre-Run Cmd/Macro  =
   (X) Data Acquisition
   ( ) Data Analysis
   ( ) Post-Run Cmd/Macro =

Method Comments:
  TO14 METHOD USING HP-DB-5 60M X 0.32MM X 1.0 FILM THICKNESS

                              END OF TOPLEVEL PARAMETERS
                              --------------------------




                              INSTRUMENT CONTROL PARAMETERS
                              -----------------------------

Sample Inlet:       GC
Injection Source:   Manual
Injection Location: Front
Mass Spectrometer:  Enabled

================================================================================
                              HP6890 GC METHOD
================================================================================

OVEN
   Initial temp:  35 'C (On)                Maximum temp:  230 'C
   Initial time:  5.00 min                  Equilibration time:  0.00 min
   Ramps:
      #  Rate  Final temp  Final time
      1  6.00       65        0.00
      2 12.00      155        0.00
      3 25.00      220        7.00
      4   0.0(Off)
   Post temp:  35 'C
   Post time:  0.00 min
   Run time:  27.10 min

FRONT INLET (UNKNOWN)                    BACK INLET ()
   Mode:  Split
   Initial temp:  200 'C (On)
   Pressure:  7.89 psi (On)
   Split ratio:  2:1
   Split flow:  3.0 mL/min
   Total flow:  7.3 mL/min
   Gas saver:  Off
   Gas type:  Helium

COLUMN 1                                 COLUMN 2
   Capillary Column                         (not installed)
   Model Number:  HP 19091J-216
   HP-5 5% Phenyl Methyl Siloxane
   Max temperature:  325 'C
   Nominal length:  59.0 m
   Nominal diameter:  320.00 um
   Nominal film thickness:  1.00 um
   Mode:  constant flow
   Initial flow:  1.5 mL/min
   Nominal init pressure:  7.90 psi
   Average velocity:  31 cm/sec

Method: TO14.M            Wed Apr 30 14:03:13 2003            Page: 1
```

Appendix III: Example Instrument parameters (continued)

```
   Inlet:  Front Inlet
   Outlet:  MSD
   Outlet pressure:  vacuum

FRONT DETECTOR (NO DET)                 BACK DETECTOR (NO DET)

SIGNAL 1                                SIGNAL 2
   Data rate:  20 Hz                       Data rate:  20 Hz
   Type:  test plot                        Type:  test plot
   Save Data:  Off                         Save Data:  Off
   Zero:  0.0 (Off)                        Zero:  0.0 (Off)
   Range:  0                               Range:  0
   Fast Peaks:  Off                        Fast Peaks:  Off
   Attenuation:  0                         Attenuation:  0

COLUMN COMP 1                           COLUMN COMP 2
   (No Detectors Installed)                (No Detectors Installed)

THERMAL AUX 2
   Use:  MSD Transfer Line Heater
   Description:
   Initial temp:  150 'C (On)
   Initial time:  0.00 min
      #  Rate  Final temp  Final time
      1   0.0(Off)

                                        POST RUN
                                           Post Time: 0.00 min

TIME TABLE
   Time       Specifier                    Parameter & Setpoint

                            7673 Injector

     Front Injector:
        Injector not configured, use these parameters if it becomes configured
        Sample Washes                2
        Sample Pumps                 4
        Injection Volume           1.0 microliters
        Syringe Size              10.0 microliters
        PostInj Solvent A Washes     4
        PostInj Solvent B Washes     0
        Viscosity Delay            0 seconds
        Plunger Speed             Fast

     Back Injector:
No parameters specified
                          MS ACQUISITION PARAMETERS

General Information
  ------- -- -----------

Tune File                : bfb.u
Acquisition Mode         : Scan

MS Information
-- -----------

Solvent Delay            : 3.80 min

EM Absolute              : False
EM Offset                : 0
Resulting EM Voltage     : 1858.8

[Scan Parameters]

Low Mass                 : 28.5
High Mass                : 260.5
Threshold                : 200
Sample #                 : 3        A/B Samples    8
Plot 2 low mass          : 28.5
Plot 2 high mass         : 260.5

[MSZones]

MS Quad                  : 106 C   maximum 200 C
MS Source                : 230 C   maximum 250 C

                             END OF MS ACQUISITION PARAMETERS

PostRun InstCntl macro(s) exist: msacq2.mac

                           END OF INSTRUMENT CONTROL PARAMETERS
```

Appendix IV: Recommended Calibration levels (based on 200 mL sample analysis)

		Level, ppb v/v[1]								
Compound	**IS Ref[2]**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
Bromochlormethane (IS#1)	NA	10	10	10	10	10	10	10	10	10
1,4-Difluorobenzene (IS#2)	NA	10	10	10	10	10	10	10	10	10
Chlorobenzene-d5 (IS#3)	NA	10	10	10	10	10	10	10	10	10
4-Bromofluorobenzene	3	10	10	10	10	10	10	10	10	10
Chlorodifluoromethane	1	-	0.2	0.4	1.0	2.5	5.0	10	20	40
Propene	1	-	-	0.4	1.0	2.5	5.0	10	20	40
Dichlorodifluoromethane	1	-	0.2	0.4	1.0	1.0	5.0	10	20	40
Chloromethane	1	-	-	0.4	1.0	1.0	5.0	10	20	40
1,2-Dichlorotetrafluoroethane	1	-	0.2	0.4	1.0	1.0	5.0	10	20	40
Vinyl Chloride	1	-	0.2	0.4	1.0	1.0	5.0	10	20	40
n-Butane	1	-	-	0.4	1.0	1.0	5.0	10	20	40
1,3-Butadiene	1	-	-	0.4	1.0	1.0	5.0	10	20	40
Bromomethane	1	-	0.2	0.4	1.0	1.0	5.0	10	20	40
Chloroethane	1	-	0.2	0.4	1.0	1.0	5.0	10	20	40
Vinyl Bromide	1	-	0.2	0.4	1.0	1.0	5.0	10	20	40
2-methyl butane	1	-	-	0.4	1.0	1.0	5.0	10	20	40
Trichlorofluoromethane	1	-	0.2	0.4	1.0	1.0	5.0	10	20	40
Acrolein	1	-	-	0.4	1.0	1.0	5.0	10	20	40
Acetonitrile	1	-	-	-	1.0	1.0	5.0	10	20	40
Acetone	1	-	-	-	-	2.5	5.0	10	20	40
Pentane	1	-	-	-	1.0	2.5	5.0	10	20	40
Isopropyl Alcohol	1	-	-	-	1.0	2.5	5.0	10	20	40
Ethyl Ether	1	-	-	-	1.0	2.5	5.0	10	20	40
1,1-Dichloroethene	1	-	0.2	0.4	1.0	2.5	5.0	10	20	40
Acrylonitrile	1	-	-	-	1.0	2.5	5.0	10	20	40
tert-butanol	1	-	-	-	1.0	2.5	5.0	10	20	40
1,1,2-Trichlorotrifluoroethane	1	-	0.2	0.4	1.0	2.5	5.0	10	20	40
Methylene Chloride	1	-	-	0.4	1.0	2.5	5.0	10	20	40
3-Chloropropene	1	-	0.2	0.4	1.0	2.5	5.0	10	20	40
Carbon Disulfide	1	-	-	0.4	1.0	2.5	5.0	10	20	40
trans-1,2-Dichloroethene	1	-	0.2	0.4	1.0	2.5	5.0	10	20	40
Methyl-t-Butyl Ether	1	-	-	-	1.0	2.5	5.0	10	20	40
1,1-Dichloroethane	1	-	0.2	0.4	1.0	2.5	5.0	10	20	40
Vinyl Acetate	1	-	-	-	1.0	2.5	5.0	10	20	40
Hexane	1	-	-	0.4	1.0	2.5	5.0	10	20	40
2-Butanone	1	-	-	-	1.0	2.5	5.0	10	20	40
cis 1,2-Dichloroethene	1	-	0.2	0.4	1.0	2.5	5.0	10	20	40
Ethyl Acetate	1	-	-	-	1.0	2.5	5.0	10	20	40
Chloroform	1	-	0.2	0.4	1.0	2.5	5.0	10	20	40
Tetrahydrofuran	1	-	-	-	1.0	2.5	5.0	10	20	40
1,1,1-Trichloroethane	1	-	0.2	0.4	1.0	2.5	5.0	10	20	40
1,2-Dichloroethane	2	-	0.2	0.4	1.0	2.5	5.0	10	20	40
Benzene	2	-	0.2	0.4	1.0	2.5	5.0	10	20	40
1-Butanol	2	-	-	-	1.0	2.5	5.0	10	20	40
Cyclohexane	2	-	-	0.4	1.0	2.5	5.0	10	20	40
Trichloroethene	2	0.1	0.2	0.4	1.0	2.5	5.0	10	20	40
Dibromomethane	2	-	-	0.4	1.0	2.5	5.0	10	20	40

[1] See section 10.3.11.
[2] Internal standard quantitation reference.

Appendix IV: Recommended Calibration levels (based on 200 mL sample analysis), continued

		Level, ppb v/v[1]								
Compound	**IS Ref[2]**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
Carbon tetrachloride	2	0.1	0.2	0.4	1.0	2.5	5.0	10	20	40
2,2,4-trimethyl pentane	2	-	-	0.4	1.0	2.5	5.0	10	20	40
n-heptane	2	-	-	0.4	1.0	2.5	5.0	10	20	40
1,2-dichloropropane	2	-	0.2	0.4	1.0	2.5	5.0	10	20	40
Bromodichloromethane	2	-	0.2	0.4	1.0	2.5	5.0	10	20	40
1,4-dioxane	2	-	-	0.4	1.0	2.5	5.0	10	20	40
Methyl Methacrylate	2	-	-	0.4	1.0	2.5	5.0	10	20	40
4-Methyl-2-pentanone	2	-	-	0.4	1.0	2.5	5.0	10	20	40
cis-1,3-Dichloropropene	2	-	0.2	0.4	1.0	2.5	5.0	10	20	40
trans-1,3-Dichloropropene	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
Toluene	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
1,1,2-Trichloroethane	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
2-Hexanone	3	-	-	0.4	1.0	2.5	5.0	10	20	40
Octane	3	-	-	0.4	1.0	2.5	5.0	10	20	40
Dibromochloromethane	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
1,2-Dibromoethane	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
Tetrachloroethene	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
Chlorobenzene	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
Ethylbenzene	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
m&p-Xylene	3	0.2	0.4	0.8	2.0	5.0	10	10	20	40
Bromoform	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
Nonane	3	-	-	0.4	1.0	2.5	5.0	10	20	40
Styrene	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
o-Xylene	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
1,1,2,2-Tetrachloroethane	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
1,2,3-Trichloropropane	3	-	-	0.4	1.0	2.5	5.0	10	20	40
Cumene	3	-	-	0.4	1.0	2.5	5.0	10	20	40
n-Propylbenzene	3	-	-	0.4	1.0	2.5	5.0	10	20	40
2-chlorotoluene	3	-	-	0.4	1.0	2.5	5.0	10	20	40
4-Ethyltoluene	3	-	-	0.4	1.0	2.5	5.0	10	20	40
1,3,5-Trimethylbenzene	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
Alpha-Methylstyrene	3	-	-	0.4	1.0	2.5	5.0	10	20	40
Decane	3	-	-	-	1.0	2.5	5.0	10	20	40
Tert-butylbenzene	3	-	-	0.4	1.0	2.5	5.0	10	20	40
1,2,4-Trimethylbenzene	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
sec-butylbenzene	3	-	-	0.4	1.0	2.5	5.0	10	20	40
1,3-Dichlorobenzene	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
Benzyl chloride	3	-	-	0.4	1.0	2.5	5.0	10	20	40
1,4-Dichlorobenzene	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
p-Cymene	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
1,2-Dichlorobenzene	3	-	0.2	0.4	1.0	2.5	5.0	10	20	40
n-butylbenzene	3	-	-	0.4	1.0	2.5	5.0	10	20	40
Undecane	3	-	-	-	1.0	2.5	5.0	10	20	40
Dodecane	3	-	-	-	1.0	2.5	5.0	10	20	40
1,2,4-Trichlorobenzene	3	-	-	-	1.0	2.5	5.0	10	20	40
Napthalene	3	-	-	0.4	1.0	2.5	5.0	10	20	40
Hexachlorobutadiene	3	-	-	-	1.0	2.5	5.0	10	20	40
1,2,3-trichlorobenzene	3	-	-	-	1.0	2.5	5.0	10	20	40

[1] See section 10.3.11.

[2] Internal standard quantitation reference

TESTAMERICA KNOXVILLE

STANDARD OPERATING PROCEDURE ATTACHMENT

TITLE: Determination of C5-C12 Total Petroleum Hydrocarbons (TPH) in Air as Octane

(SUPERSEDES: KNOX-MS-0001, Attachment A, Revision 1)

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1. Scope and Application

1.1. This attachment describes additional procedures that are employed in order to quantitate total petroleum hydrocarbon (TPH) as octane in the C5-C12 range in samples analyzed by TO-14A or TO-15. See Appendix I, Table 1 for the list of target analytes and reporting limits.

2. Summary of Method

2.1. The TPH concentration is determined from all peak areas in the RIC summed from C5-C12 minus areas contributed by the surrogates and internal standards in the RIC. The final result is quantitated against the octane response factor obtained from the RIC in the TO-14/15 initial calibration.

2.2. The compounds analyzed by this method are listed in Appendix 1, Table 1.

3. Definitions

3.1. RIC: Reconstructed Ion Chromatogram

4. Interferences

4.1. Interferences in the RIC on the internal standard can cause a bias in the quantitation of TPH. The analyst must be intimately familiar with the internal standard and surrogate peak shape, retention time, and uninterfered height/area in order to determine matrix bias. Inspection of the RIC is required to ensure that there is no interference to the internal standards/surrogate in order to make appropriate decisions to remove or include the areas for calculation.

4.2. Non-TPH peaks can contribute to a bias in the results. Since it is not practical to examine every peak in the chromatogram, this method reports all peaks between C5 and C12. However, the advantage of TPH by GC/MS is that it allows the operator to tentatively identify extraneous peaks that could inflate the TPH values that may or may not be normally found in hydrocarbon mixtures. If the operator is aware of non-TPH peaks are present (e.g. chlorinated solvents), the operator may elect to remove the area of the non-TPH peaks to provide a lower-biased result. For example, a non-TPH peak may be present that would be cause to dilute a sample; in this case the non-TPH peak may be excluded and the sample would not have to be diluted.

4.3. Instances of obvious bias must be narrated in the project case narrative.

5. Safety

5.1. There are no additions to this section of the SOP.

6. Equipment and Supplies

6.1. There are no additions to this section of the SOP.

7. Reagents and Standards

7.1. Unleaded Gasoline Composite, Restek Catalogue Number 30205 or equivalent, 50000 ug/mL in methanol. (Other stock standard concentrations may be used.)

8. Sample Collection, Preservation and Storage

8.1. There are no additions to this section of the SOP.

9. Quality Control

9.1. Internal/Surrogate Standards

9.1.1. Internal standard 1,4-difluorobenzene RIC area is used for the quantitation of TPH. In addition to the $\pm$ 20 second RT time criteria in the SOP, the samples' internal standard RIC area is compared to the daily calibration's internal standard RIC area. The limit is $\pm$ 40% D. If the recovery is outside 40%, the internal standard RIC must be inspected for interferences. If in the TO-14A/TO-15 analysis (which uses the internal standard quantitation ion for calculation) the internal standard recovery is within control, then the analysis is within control and matrix interferences are likely on the RIC. If interferences are present, then the data is reported and narrated that matrix interferences are present which would bias the TPH results.

9.1.2. Surrogate recovery is calculated and reported from the quantitation ion analysis described in the SOP.

9.2. Laboratory Control Standard (LCS)

9.2.1. The LCS analytes of interest is the sum of the areas of octane, 2,2,4-trimethylpentane, decane, and dodecane from the RIC of the daily calibration verification standard. The sum of these individual peak

areas are quantitated against the response factor of octane. The recovery of TPH LCS must be within 50-150%.

10. Calibration and Standardization

10.1. Initial Calibration

10.1.1. Using the initial calibration standards and limits described in the SOP, octane is quantitated based on the response from the RIC. (Upon client request hexane or another compound can be used). See Appendix IV of the TO-14A & TO-15 SOP for the recommended calibration amounts.

10.1.1.1. If the %RSD of the TPH initial calibration is > 30%, but the %RSD in the TO-15 calibration met the TO-15 criteria, analysis may proceed. Calibration points for the TPH compound may be dropped to achieve better linearity (see 10.3.11 of the TO-15 SOP for rules on dropping calibration points). The calibration model may be changed to linear or weighted linear as long as the r2 is $\geq$ 0.990 and the intercept is < RL. Quadratic fit should not be used

10.1.2. The response factor of octane is obtained and entered into the appropriate Target processing methods for TPH in air.

10.2. Daily Calibration Verification

10.2.1. Using the daily calibration standard and limits described in the SOP, octane is quantitated based on the response from the RIC.

11. Procedure

11.1. The area of TPH is the sum of all the peaks in the RIC from pentane (- 0.05 minutes) to dodecane (+ 0.05 minutes) minus all peak areas of internal standards and surrogates from the TO-14A & TO-15 analysis.

11.2. Analyst must inspect the chromatograms for proper integration and interferences.

11.3. Any individual TPH area that exceeds the area of octane in the high point of the calibration curve is considered over range and must be diluted by procedures described in the SOP. Peaks that are determined not to be part of TPH (e.g.

chlorinated solvents) are not reason to dilute the sample and the non-TPH area is subtracted from the total area for calculation.

11.4. Samples that are initially diluted to obtain on-scale TO-14A/15 results are not analyzed more concentrated if TPH are not detected above the reporting limit.

11.5. If TO-15 analytes are < 20% of the calibration range but reanalysis of the sample to bring the analytes in the upper half of the calibration range would cause the TPH value to be over the calibration range, the sample does not need to be analyzed more concentrated.

12. Data Analysis and Calculations

12.1. Response Factor (RF) octane to calculate TPH:

$$RF = \frac{Ax * Cis}{Ais * Cx}$$

where:

Ax	=	area of octane from the RIC.
Ais	=	area of 1,4-difluorobenzene for the internal standard from the RIC.
Cx	=	concentration amount of octane in ppb v/v.
Cis	=	amount of the internal standard (1,4-difluorobenzene) in ppb v/v.

12.2. TPH concentration in samples is calculated using the average response factor of octane from the initial calibration of the RIC.

In the formulas presented in the SOP, replace the following

R_x = TPH RIC area of pentane (C5) -0.05 min. to dodecane (C12) + 0.05 min.
R_{is} = internal standard RIC area
RF = Response factor of octane obtained from initial calibration RIC

12.3. Estimated values below the reporting limit are not reported for TPH.

13. Method Performance

13.1. Reporting Limit (RL) - An RL standard must be analyzed for TPH on each instrument per year. A RL standard of gasoline of a known concentration in a

canister must be no greater than 2X the reporting limit. The reporting limit standard must be at least 3X the area of the daily method blank.

13.2. Initial Demonstration of Capability – Each analyst must perform an initial demonstration of capability (IDOC) to performing the analysis independently. The IDOC is determined by analyzing four replicates of gasoline of a known concentration in a canister as detailed in TestAmerica Knoxville SOP KNOX-QA-0009. Recovery limits must be 50-150% and RSD must be less than or equal to 25%.

14. Pollution Prevention

14.1. There are no additions to this section of the SOP.

15. Waste Management

15.1. There are no additions to this section of the SOP.

16. References

16.1. Agency for Toxic Substances & Disease Registry, http://www.atsdr.cdc.gov/mhmi/mmg72.html

17. Miscellaneous

17.1. Appendix I: Table 1 Target Analyte

Appendix I: Table 1: Target Analytes

CAS NUMBER	TestAmerica Knoxville Compounds	MOLECULAR WEIGHT (ng/nmole)	conversion factor for ug/m3 = MW/24.45	200 mL REPORT-ING LIMIT (ppb(v/v)	200 mL REPORT-ING LIMIT (ug/m3)	500 mL REPORT-ING LIMIT (ppb(v/v)	500 mL REPORT-ING LIMIT (ug/m3)	SUGGESTED ION
n/a	TPH (as octane)[1]	108[2]	4.41718	10	44	4	17	RIC

1 – TPH may also be reported based on another reference peak, e.g., TPH as hexane.
2 – The average molecular weight of gasoline is taken from http://www.atsdr.cdc.gov/mhmi/mmg72.html

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Canton

SOP No. NC-WC-018, Rev. 3
Effective Date: 05/10/13

Title: TOTAL ORGANIC CARBON (TOC) ANALYSIS FOR NON-WATERS

[Method: Walkley-Black]

Approvals (Signature/Date):

[redacted]	05/09/13	[redacted]	05/09/13
Technology Specialist	Date	Health & Safety Coordinator	Date
[redacted]	05/09/13	[redacted]	05/09/13
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. NC-WC-018, Rev 2.4, dated 09/30/10

Facility Distribution No. _____ **Distributed To: _____**

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Total Organic Carbon in oils, sludge, soil, and sediment samples. It is based on Methods of Soil Analysis, Walkley-Black. The working linear range is 1000 to 15,000 mg/kg.

1.2. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory.

2. SUMMARY OF METHOD

2.1. An aliquot of a solid sample is treated with excess potassium dichromate and concentrated sulfuric acid. After treatment, the solution is backtitrated with ferrous sulfate to determine the amount of dichromate reduced during digestion.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. This method uses strong oxidizers, which can cause severe burns and tissue destruction.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there

are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 mg/m^3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.7. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.8. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.9. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Buret: 10 mL Class A

6.2. Top loading balance: capable of weighing to ± 0.01 g

6.3. Graduated cylinders: various, Class A

6.4. Volumetric pipettes: various , Class A

6.5. Erlenmeyer flasks: various

6.6. Whatman #4 filter paper

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Sulfuric Acid ($H_2 SO_4$): concentrated, Tracepur grade

7.1.2. Ferroin indicator, purchased

7.1.3. Potassium Dichromate ($K_2Cr_2O_7$): primary standard grade

7.1.4. 1N Potassium Dichromate Solution: Accurately weigh 49.04 g of potassium dichromate (dried overnight at 105°C) in a liter volumetric flask and dilute to volume. Store in amber bottle and refrigerate. Replace after six months.

7.1.5. Ferrous Sulfate ($FeSO_4 \bullet 7 H_2O$): reagent grade

7.1.6. 0.5 N Ferrous Sulfate Titrant: Accurately weigh 140 g of $FeSO_4 \bullet 7H_2O$ into a 1 liter volumetric flask and dissolve with 500 mL reagent water. Carefully add 15 mL of concentrated sulfuric acid and allow to cool. Dilute to volume with reagent water. Store in amber bottle and refrigerate.

7.2. Standards

7.2.1. Laboratory Control Sample

7.2.1.1.Potassium Acid Phthalate (KHP), 0.05N (4800 mg/L)

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Samples are stored in a glass container at 4°C ± 2°C.

8.2. Samples are not chemically preserved. In lieu of no guidance, holding time is based on water requirements.

8.3. The holding time is 28 days from sampling to analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) that are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank (MB)

9.2.1. One MB must be processed with each preparation batch. The MB consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.2. An MB consisting of 2.5 g Ottawa sand and 200 mL reagent water is prepared and analyzed with each analytical batch of samples.

9.2.3. Corrective Action for Method Blanks

9.2.3.1. If the analyte level in the MB exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.3.2. If there is no analyte greater than the RL in the samples associated with an unacceptable MB, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. A midrange LCS using 1 mL of Potassium Acid Phthalate (KHP), 0.05N spiked onto 2.5g of Ottawa sand is prepared and analyzed with each batch of samples.

9.3.3. Corrective Action for LCS

9.3.3.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.3.2. The only exception is that if the LCS recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

9.3.3.3. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Sample Duplicate (DU)

9.4.1. A DU is a second aliquot of an environmental sample, taken from the same sample container when possible, that is processed with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. The sample and DU results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample/DU precision results are not necessarily representative of the precision for other samples in the batch.

9.4.2. DUs are performed at a frequency of 10% or one per batch which ever is more frequent and must meet laboratory-specific limits for precision.

9.5. Control Limits

9.5.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.5.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMs.

9.6. Method Detection Limits (MDLs) and MDL Checks

9.6.1. MDLs and MDL checks are established by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.6.2. MDLs are easily accessible via LIMs.

9.7. Nonconformance and Corrective Action

9.7.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action.

10. CALIBRATION AND STANDARDIZATION

10.1. The ferrous sulfate titrant is standardized daily as follows.

10.1.1. Pipette 10.0 mL of 1.00 N potassium dichromate solution into a 250 mL Erlenmeyer flask and add 90-mL reagent water.

10.1.2. Carefully add 30 mL of concentrated sulfuric acid and allow cooling completely. **See note in section 5.4.**

10.1.3. Add 4-5 drops of ferroin indicator.

10.1.4. Titrate with 0.5 N ferrous sulfate titrant to a reddish-brown endpoint or to the first color change after reaching an emerald-green color.

10.1.5. Calculate the normality using the following equation.

$$N = \frac{10}{mL\ ferrous\ sulfate}$$

10.1.6. Repeat steps 10.1.1 through 10.1.5 two more times.

10.1.7. The average of the triplicate standardization is used.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of QA, Operations Supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. Physical Preparation

11.3.1.1. Mix the sample thoroughly before selecting a portion for analysis.

11.3.1.2. Discard any foreign objects such as sticks, leaves, and rocks.

11.3.2. Analytical Preparation

11.3.2.1. Weigh an aliquot of soil of 2.50 g to the nearest 0.01 g (use less sample if TOC is known to be high). Record the weight in LIMS.

11.3.2.2. Place sample in a 500 mL Erlenmeyer flask, and add 10.0 mL of 1 N potassium dichromate with a Class A glass pipette.

11.3.2.3. Under a hood, carefully add 20 mL of concentrated sulfuric acid and gently swirl for one minute. **See note in section 5.4.**

11.3.2.4. Allow sample to cool for about 30 minutes.

11.3.2.5. Add 200 mL of reagent water and swirl to mix. If necessary, filter sample through Whatman #4 filter.

11.4. Sample Analysis Procedure

11.4.1. Add 4-5 drops ferroin indicator.

11.4.2. Using a 10 mL Class A glass burette, titrate with 0.5 N ferrous Sulfate Solution to a reddish-brown endpoint or first color change after reaching an emerald-green color.

11.4.2.1. If the digestate of the sample is already green or reddish-brown after the addition of the ferroin indicator or if less than 2 mL of titrant is used, the sample needs to be re-extracted with a smaller sample amount.

11.4.3. Document the amount of titrant in LIMS.

11.5. Analytical Documentation

11.5.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.5.2. Record all standards and reagents in the LIMS Reagents Module. All standards and reagents are assigned a unique number for identification.

11.5.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4. Record all sample results and associated QC directly into LIMS during analysis. Level I and Level II review is performed in LIMS.

12. DATA ANALYSIS AND CALCULATIONS

12.1. **Total Organic Carbon, mg/kg =**

$$\left[\frac{\textbf{[(10)(N Potassium Dichromate) - (mL Ferrous Sulfate) (N Ferrous Sulfate)]}}{\textbf{Weight of Soil (g)}} \textbf{ x 3000}\right] \times \textbf{1.3}$$

Where: 1.3 = Correction factor recommended in method

NOTE: The 1.3 correction factor should not be used when calculating the MB or LCS. See section 17.3.

12.2. $\textbf{TOC, \%} = \frac{\textbf{mg / kg}}{\textbf{10,000}}$

12.3. $\textbf{LCS, \%} = \frac{\textbf{TOC, \%}}{\textbf{1920}} \textbf{ x 100}$

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual for "Waste Management and Pollution Prevention".

15.2. Waste Streams Produced by the Method

15.2.1. The following waste streams are produced when this method is carried out.

15.2.1.1. Aqueous acidic material from the digestion process and titrant standardization: This sample waste is collected in the laboratory in a designated waste container identified as "Acid Waste".

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by annual refresher training.

16. REFERENCES

16.1. References

16.1.1. Methods of Soil Analysis, 1982 Second Edition Method 29-3.5.2 Walkley-Black Procedure

16.1.2. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.4. Corporate Quality Management Plan (CQMP), current version

16.1.5. Revision History

Historical File:		Revision1: 01/10/97		
		Revision2: 02/27/97		
		Revision 2.1: 03/30/01		
		Revision 2.2: 10/26/04		
		Revision 2.3: 05/30/08		
		Revision 2.4: 09/30/10		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-016

16.2.6. Standards and Reagents, NC-QA-017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. The lower reporting limit (RL) for undiluted samples is 1000 mg/kg.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Troubleshooting guide

17.2.1. When interferences as described in Section 4 are encountered or suspected, treat the sample as specified in that section.

17.2.2. If a high level of TOC is suspect (black sample), a smaller amount will be required.

17.3. Method Deviations

17.3.1. The 1.3 correction factor is not applied to the MB and LCS since no carbon is present or spiked in a solid form.

Appendix E

Laboratory Sample Preparation Standard Operating Procedures

TestAmerica
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Canton

SOP No. NC-OP-037, Rev. 3
Effective Date: 04/05/13

Title: CONTINUOUS LIQUID / LIQUID EXTRACTION OF ORGANIC COMPOUNDS FROM WATERS BASED ON METHOD SW846 3520C AND 600 SERIES

[Methods: SW846 3520C and 600 Series]

Approvals (Signature/Date):

[redacted]	04/04/13	[redacted]	04/05/13
Technology Specialist	Date	Health & Safety Coordinator	Date
Rebecca Strait	04/04/13	[redacted]	04/04/13
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. NC-OP-037, Rev 2, dated 02/05/13,

Facility Distribution No. _____ **Distributed To: _____**

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1. SCOPE AND APPLICATION

1.1 This SOP describes procedures for preparation (extraction) of semivolatile organic analytes in aqueous, and TCLP leachate, matrices for analysis by Gas Chromatography (GC) and Gas Chromatography/ Mass Spectrometry (GC/MS) using Continuous Liquid/Liquid Extraction. The procedures are based on SW846 and 600 series methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.1.1. Extraction procedures for the following determinative methods are covered: 8081A, 8081B, 8082, 8082A, 8270C, 8270D, 8015B, 8015C, 608, and 625.

1.1.2. The extraction procedures here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

2. SUMMARY OF METHOD

2.1. Continuous Liquid/Liquid Extraction

2.1.1 A measured volume of sample (typically 1 liter, or 250 mL for reduced volume extraction requiring large volume injection) is placed into a continuous liquid/liquid extractor, adjusted if necessary, to a specific pH, and extracted with the appropriate solvent for 18-24 hours.

2.2. Concentration

2.2.1 Procedures are presented for drying and concentration of the extract to final volume for analysis.

3. DEFINITIONS

3.1. Definitions of terms and acronyms used in this SOP may be found in the glossary of the TestAmerica Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated must be removed and discarded, other gloves must be cleaned immediately.

5.3. The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds must be prepared in the hood.

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3 2 mg/m^3-Ceiling	This material will cause burns if comes into contact with the skin or eyes. Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.

Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain, and severe tissue burns. Can cause blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride must be conducted in a fume hood with the sash closed as far as the operations will permit. If more than 500 mL of methylene chloride is spilled, evacuate the area until the area has been cleaned by EH&S.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus and glass separatory funnels have ground glass joints, which can become stuck. Technicians must use Kevlar or other cut/puncture-resistant gloves when separating stuck joints.

5.9. 3520 Extraction Continuous Liquid/Liquid

5.9.1. All personnel are to ensure liquid-liquid area is clear of unnecessary items. Heating mantles used with liquid-liquid extractions generate temperatures that could ignite some materials that come in contact with the heating mantles.

5.9.2. Ensure all solvents are away from liquid-liquid extractor. Increased temperatures near solvents can cause the pressure in the containers to increase.

5.9.3. Ensure all boiling flasks have cooled to room temperature before disconnecting liquid-liquid bodies from boiling flasks to prevent any burns.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware must be cleaned per Glassware Washing, SOP NC-QA-014.

6.2. Equipment and supplies for extraction procedures:

EQUIPMENT AND SUPPLIES	CLLE	Conc
pH Indicator paper, ranges: 0-14, 7.5-14, 0-6	√	
Graduated cylinder: 1 liter. (other sizes may be used as needed)	√	√
Methylene chloride collection tank	√	
Initial volume template	√	
Solvent dispenser pump or 100 mL graduated cylinder		√
Continuous liquid / liquid extractor	√	
Round or flat bottom: 250	√	
Boiling chips: contaminant-free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent)	√	√
Cooling condensers	√	
Heating mantle: rheostat controlled	√	
Auto-timer for heating mantle	√	
Beakers: 250 & 400 mL, graduated	√	√
Kuderna-Danish (K-D) apparatus: 500 mL		√
Concentrator tube: 10 mL, attached to K-D with clips		√
Snyder column: three-ball macro		√
Water bath: heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.		√
Vials: glass, 2 / 2.5/ 40 mL capacity with Teflon®-lined screw-cap		√
Nitrogen blowdown apparatus		√
Nitrogen: reagent grade.		√
Culture tubes: 10 mL, 16 mmx100 mm		√
Microliter pipette, syringe 1 mL	√	
Glass wool	√	
Funnel: 75 X 75 mm	√	√
Disposable pipettes, 5 ¾ in, and 9in.	√	√
Aluminum foil	√	√
Paper towels	√	√

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better unless otherwise specified.

REAGENTS	CLLE	Conc
Sodium hydroxide (NaOH), pellets: reagent grade	√	
Sodium hydroxide solution, 10 N: dissolve 40 g of NaOH in reagent water and dilute to 100 mL.	√	
Sulfuric acid (H_2SO_4), concentrated: reagent grade	√	
Sulfuric acid (1:1): carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.	√	
Hydrochloric acid (HCl)	√	
Organic-free reagent water	√	
Sodium sulfate (Na_2SO_4), granular, anhydrous: purify by heating at 400°C a minimum of two hours	√	√
Extraction / exchange solvents: methylene chloride, hexane, acetonitrile, acetone, pesticide quality or equivalent	√	√
Acetone, methylene chloride: used for cleaning	√	√
Sodium Chloride (NaCl) crystal	√	

7.2. Standards

7.2.1. Stock Standards

7.2.1.1 Stock standards are purchased as certified solutions. Standards shall be stored according to manufacturer's instructions. All stock standards must be protected from light. Stock standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampoule is opened if purchased). Standards must be allowed to come to room temperature before use.

7.2.2. Surrogate Spiking Standards

7.2.2.1 Prepare or purchase surrogate spiking standards at the concentrations listed in Table 5. Surrogate spiking standards are purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light or stored according to manufacturer's instructions. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.3. Matrix Spiking and Laboratory Control Spiking Standards

7.2.3.1 The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Table 6. Spiking standards are purchased or

prepared as dilutions of the stock standards.

7.2.3.2 Spiking solutions must be refrigerated and protected from light or stored according to manufacturer's instructions. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.4 See SOP NC-QA-017 for additional information on Standards and Reagents.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1 The holding time for aqueous samples is seven days from sampling to extraction.

8.3.2 For TCLP leachates, the holding time is 14 days from sampling to the leach process. The extraction holding time seven days from when the TCLP Leach tumbling has been completed, excluding the filtration step, to the extraction step.. If the filtration step requires extended times, this time counts as part of the seven-day holding time.

8.3.3. Analysis of the extracts is completed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

9.1.1. The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS, and a matrix spike / matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS / MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD. See Policy QA-003 for further definition of the batch.

9.2. Sample Count

9.2.1. Laboratory-generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count. Field samples are included.

9.3. Method Blank

9.3.1. A method blank consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. See Table 3 for the appropriate amount of surrogate to use for each analytical method. The method blank is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data.

9.3.2. Aqueous Method Blanks use 1000 mL (or 250 mL for reduced volume extraction) of reagent water spiked with the surrogates. The method blank goes through the entire analytical procedure.

9.3.3. TCLP method blanks use 250 mL of leachate fluid spiked with the surrogates. SPLP method blanks use 1000 mL of leachate fluid spiked with the surrogates. The leachate may optionally be diluted to 1000 mL with reagent water. The method blank goes through the entire analytical procedure.

9.4. Laboratory Control Sample (LCS)

9.4.1. Laboratory Control Samples are well-characterized laboratory-generated samples used to monitor the laboratory day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure.

9.4.2. The LCS is made up in the same way as the method blank (see Sections 9.3.1 through 9.3.3), but spiked with the LCS standard and the surrogates. See Tables 3 and 4 for the appropriate amount of spike to use for each analytical method.

9.5. Surrogates

9.5.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.5.2. Each applicable sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. See Table 3 for the appropriate amount of surrogate spike to use for each analytical method.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1. A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and matrix spike. See Tables 3 and 4 for the appropriate amount of spike to use for each analytical method.

9.7. Initial Demonstration of Capability

9.7.1. The initial demonstration and method detection limit studies described in Section 13 must be acceptable before analysis of samples may begin.

9.8 Control Limits

9.8.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.9 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMs

9.10 Method Detection Limits (MDLs) and MDL Checks

9.10.1 MDLs and MDL Checks are established by the laboratory as described in SOPs

9.10.2 MDLs are accessible via LIMs.

9.11 Nonconformance and Corrective Action

9.11.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action.

10. CALIBRATIONAND STANDARDIZATION

10.1. On a weekly basis, measure the appropriate volume of solvent into the appropriate size glass vial using a gastight syringe that is manufactured to a certified volume delivery tolerance of ± 0.01 mL. The "standard" glass vial is sealed, and the meniscus is noted by marking a line on the bottle. The glass vials containing the sample extracts are then compared against the "standard" glass vial to ensure the final volume is consistently 1.0 ± 0.01 mL. A log is kept of the glass vial lot number and preparation date.

11. PROCEDURE

Refer to SOP NC-QA-016 for information on DoD samples.

11.1. Procedural Variations

11.1.1 Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance memo and approved by a supervisor. The Nonconformance memo will be filed in the project file. Procedural variations are not allowed for Ohio VAP projects.

11.2. Continuous Liquid/Liquid Extraction from Water Samples

11.2.1 Remove surrogate and matrix spiking solutions from refrigerator and allow to return to room temperature.

11.2.2 Assemble the apparatus. Add approximately 250 mL of methylene chloride (or approximately 100 mL for reduced volume extractor bodies) to the extractor body. Add three to five boiling chips to the round-bottom distilling flask. Label the flask with a LIMS ID label.

11.2.3 Measure the initial sample pH with wide-range pH by inserting a disposable pipette into the sample, and placing a drop of sample onto the wide range pH paper. Record on the extraction benchsheet. pH will be entered manually into LIMS during level review. (**what "level" review?)**

11.2.4 Measure the initial volume using the volume template. Place the template next to the sample bottle and read the volume marking from the template. Record this volume on the benchsheet. Volumes will be entered manually into LIMS during level review. Prepare a method blank, LCS, and MS/MSD for each batch as specified in Section 9 of this SOP. See Tables 3 and 4 for the appropriate amount of spike to use for each analytical method. Use 1 L of reagent water for method blanks and LCS. If the sample cannot be prepared using continuous liquid/liquid extraction due to matrix, a waste dilution may be required. Refer to Section 11.3 for the waste dilution procedure.

11.2.5 Use 250mL of leachate for TCLP semivolatiles and TCLP pesticides. Use 1000 mL of leachate for SPLP semivolatiles and SPLP pesticides. Dilute to about 1 liter with reagent water.

11.2.6 For a TCLP method blank and LCS, measure 250 mL of the buffer solution in a beaker and transfer to the continuous liquid/liquid extractor. Dilute to about 1 liter with reagent water. For an SPLP method blank and LCS,, measure 1000 mL of the buffer solution using the volume template and transfer to the continuous liquid/liquid extractor. No dilution with reagent water is required.

11.2.7 Less than one liter of sample may be used for highly contaminated samples, or if the reporting limit can be achieved with less than one liter of sample. In this event, dilute the sample to about 1 liter with reagent water. This must be documented with a Non-Conformance Memo.

11.2.8 Add reagent water to the extractor body until approximately 150 mL (approximately 50mL for reduced volume preps) of methylene chloride is pushed over into the round-bottomed flask to ensure proper operation and solvent cycling. Prime the extractor using reagent water.

11.2.9 The method blank and samples are spiked with the surrogates, the LCS and matrix spikes with the surrogates, and matrix spiking solutions. All samples are spiked in the original sample bottle.

11.2.10 Pour the sample into the extraction vessel. Adjust sample pH as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH, as necessary. Recheck the sample with pH paper. Record adjusted pH, spiking volumes and standard numbers on the benchsheet. Return spiking solutions to the refrigerator as soon as possible. Attach cold condenser (about 10°C). Turn on heating mantle. Inspect joints for leaks once solvent has begun cycling. Extract for 18-24 hours (24 hours required for 600 series).

11.2.11 If extraction at a secondary pH is required (see Table 1), turn off the heating mantle and allow the extractor to cool. Detach the condenser and adjust the pH of the sample in the extractor body to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure by inserting a disposable pipette into the sample, and placing a drop of sample on the pH paper. Record the adjusted pH on the benchsheet. Re-attach the condenser, and turn on the heating mantle. Extract for 18-24 hours.

11.2.12 Turn off the heating mantle and allow the extractor to cool.

11.2.12 Cover with aluminum foil and refrigerate if the extract is not concentrated immediately. Refer to Section 11.4 for concentration.

11.3 Concentration

According to the type of sample, different solvents and final volumes will be required. Refer to Table 2 for the appropriate final volumes and concentrations.

11.3.1 Kuderna-Danish (KD) Method

11.3.1.1 Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube to the 500 mL K-D flask. Label the CT and K-D. Transfer the sample to the labeled K-D flask, filtering Continuous Liquid/Liquid and Soxhlet samples through funnels filled with sodium sulfate. Rinse the funnel with 20-30 mL of methylene chloride to complete the quantitative transfer.

11.3.1.2 Add one or two clean boiling chips to the KD flask and attach a three-ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column. (This is important to ensure that the balls are not stuck, and the column will work properly). Attach to the KD flask.

11.3.1.3 Place the KD apparatus on a water bath (90-98°C) so the tip of the concentrator tube is submerged. The water level must not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter; but the chambers should not flood.

11.3.1.4 Concentrate to 15-20 mL. If the determinative method requires a solvent exchange, add the appropriate exchange solvent to the top of the Snyder Column, and then continue the water bath concentration back down to 5-8 mL. Refer to Table 2 for details of exchange solvents and final volumes. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.3.1.5 Remove the KD apparatus from the water bath and allow to cool for a minimum of 10 minutes. If the level of the extract is above the level of the concentrator tube joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

11.4 Nitrogen Evaporation to Final Concentration

11.4.1 Transfer the CT to the evaporation apparatus.

11.4.2 Place the tube in a warm water bath that is at least 5°C below the boiling temperature of the solvent being evaporated and evaporate the solvent using

a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but must not create splattering of the extract.

Boiling points of commonly used solvents are:

Methylene chloride	40°C
Acetone	56°C
Hexane	69°C
Acetonitrile	82°C
Toluene	111°C

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.4.3 Refer to Table 1 to determine the final volume needed for a specific test method. Evaporate to slightly less than the required final volume.

11.4.4 Quantitatively transfer the extract to the appropriate final container and dilute to the appropriate final volume using the "standard" glass vial noted in Section 10.1. Cap the sample and affix the appropriate label. The sample is now ready for analysis.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

11.5 Analytical Documentation

11.5.1 Record all analytical information in LIMS, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2 All standards are logged into the LIMS standards and reagents module. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3 Sample information and associated QC are entered into the LIMs. Technical reviews are done in LIMS.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

13.1.1. Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which must contain all the analytes of interest. The spiking level must be equivalent to a mid-level calibration. (For certain tests, more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

13.1.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.1.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs. See SOPs NC-GC-038, NC-MS-018, NC-MS-003, and NC-GC-007 for detailed information on the determinative methods.

13.2. Training Qualification

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.2.2 Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. The following waste streams are produced when this method is carried out.

15.2.1. Extracted aqueous samples contaminated with methylene chloride. This tank is then periodically rolled to the Tank Room, the pH is verified, contents are neutralized with sodium bicarbonate, pH re-verified, and Dichloromethane waste drained into a waste drum located outside the building. The wastewater is discharged to the POTW.

15.2.2. Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride/acetone or acetone/hexane from the extract drying step. These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.3. **Assorted flammable solvent waste from various rinses**. These wastes are put into the halogenated/non-halogenated 25 gallon solvent waste container located under the fume hood in extractions.

15.2.4. **Methylene chloride waste from various rinses:** These wastes are disposed of in the liquid-liquid separation unit.

15.2.5. **Hexane- Hexane waste:** These samples are to be disposed in the flammable waste.

15.2.6. **Waste Hexane in vials.** These vials are placed in the vial waste located in the GC prep laboratory.

15.2.7. **Waste Methylene Chloride sample vials**. These vials are placed in the vial waste located in the GC prep laboratory.

15.2.8. Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCBs must be segregated into their own waste stream. PCB wastes are collected in one of three waste streams, solid PCB, liquid PCB, and PCB vial waste.

15.2.9. Solvent Recovery System Waste. Methylene Chloride waste from the Solvent Recovery System is collected and disposed of in the liquid-liquid separation unit. Acetone/Methylene Chloride waste from this system is disposed of in the flammable waste containers located in the laboratory.

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/ChemicalMethods, SW846, 3rd Edition, Final Update III (December 1996). Sections 3500B, 3520C, and 3580A

16.1.2 Federal Register - EnvironmentalProtection Agency, 40 CFR, Part 136, Volume 49, No. 209, October 26, 1984, Method 625

16.1.3 EPA 600, Methods for Chemical Analysis of Water and Wastes, Method 608

16.1.2. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.3. TestAmerica Corporate EnvironmentalHealth and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.4. Corporate Quality Management Plan (CQMP), current version

16.1.5. Revision History

Historical File:		Revision3.4: 10/16/98		Revision 0: 03/12/08 (NC-OP-032)
(formerly CORP-OP-0001NC)		Revision 3.5: 04/22/99		Revision 1: 01/07/09 (NC-OP-032)
		Revision 3.6: 05/13/99		Revision 0: 03/03/11 (NC-OP-037)
		Revision 3.7: 03/20/01		Revision1: 04/24/12
		Revision 3.8: 05/23/01		Revision2: 02/05/13
		Revision 3.9: 04/22/02		
		Revision 4.0: 02/04/03		
		Revision 4.1: 10/07/03		
		Revision 4.2: 01/30/06		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5. Supplemental Practices for DoD Project Work SOP, NC-QA-016

16.2.6. Gas Chromatographic Analysis based on Method 8000B, 8021B, 8081A, 8081B, 8082, 8082A, 8151A, 8015B, 8015C, and 615, NC-GC-038

16.2.7. GC/MS Analysis based on Method 8270C and 8270D, NC-MS-018

16.2.8. Analysis of Pesticides and PCBs by EPA Method 608, NC-GC-007

16.2.9. GC/MS Semivolatile Organic Compounds Capillary Column Technique Based on EPA Method 625, NC-MS-003

16.2.10. Standards and Reagents, NC-QA-017

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.1.2. Spiking levels for method 608 have been reduced by a factor of ten to bring the levels within the normal calibration range of the instrument.

17.2. Tables

TABLE 1
Liquid /Liquid Extraction Conditions

Determinative Method	Initial Ext. pH	Secondary Ext. pH
BNA	Acid ext; 1-2 or Base ext; 11-12	Acid ext; 1-2 or Base ext; 11-12
Pesticide/PCB	5-9	None
TPH	As received	None

[1] If the laboratory has validated acid only 8270 extraction for the target compound list required then the base extraction step may be omitted. The required validation consists of a four-replicate initial demonstration of capability and a method detection limit study (see Section 13). Additionally, either of the base or acid fractions of Method 8270 can be run first.

TABLE 2
Final Volumes and Exchange Solvents

Type	Exchange Solvent for Analysis	Final Volume for Analysis in mL
BNA	N/A	2.0 mL
PCB	Approximately 18 mL hexane – water	5.0 for H2O 2.0 for H2O*
Pesticides	Approximately 18 mL hexane	5.0 for H2O 2.0 for H2O*
Pesticides/TCLP	Approximately 18 mL hexane	3.0 mL
BNA – SIM	N/A	2.0 mL 5.0 mL for reduced volume preps
TPH	N/A	5 mL

* Michigan work requires a final volume of 2 mL.

Note: Different final volumes may be necessary to meet special client reporting limit requirements.

TABLE 3
Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume (mL)
BNA	20 ppm BNA	1.0
BNA– SIM	100/150 ppm BNA	0.2 / 0.02
Pesticides	0.2 ppm DCB/TCX	1.0
TPH	40ng Nonane (C9)	1.0
PCB	0.2 ppm DCB/TCX	1.0

* Note: surrogate spiking levels are adjusted for reduced volume preps which utilize large volume injection.

TABLE 4 Matrix Spike and LCS Solutions		
Analyte Group	**Matrix Spike Solution ID**	**Volume (mL)**
BNA	20 ppm BNA All-Analyte Spike and Restek Spike	1.0
BNA– SIM	100 ppm BNA All-Analyte Spike and Restek Spike	0.2 / 0.02
Pesticides	Pest NPDES Spike	1.0
Pesticides/ TCLP	Pest TCLP Spike	1.0
PCB	10 ppm PCB Spike	1.0
TPH	See Spike List – Table 6	1.0

* Note: surrogate spiking levels are adjusted for reduced volume preps which utilize large volume injection.

TABLE 5 Surrogate Spike Components		
Analyte Group	**Compounds**	**Conc. (µg/mL)**
BNA	2-Fluorobiphenyl	100
	Nitrobenzene-d5	100
	p-Terphenyl-d14	100
	2-Fluorophenol	150
	Phenol-d6	150
	2,4,6-Tribromophenol	150
	1,2-Dichlorobenzene-d4	100
	2-Chlorophenol-d4	150
Pesticides PCB	Decachlorobiphenyl	0.2
	Tetrachloro-m-xylene	0.2

TPH	Nonane (C9)	40.0

TABLE 6
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
BNA	Acenaphthene	100
	4-Chloro-3-Methylphenol	100
	2-Chlorophenol	100
	1,4-Dichlorobenzene	100
	2,4-Dinitrotoluene	100
	4-Nitrophenol	100
	Pentachlorophenol	100
	Phenol	100
	Pyrene	100
	1,2,4-Trichlorobenzene	100
	1,4-Dichlorobenzene	100
	2,4-Dinitrotoluene	100
	Hexachlorobenzene	100
	Hexachlorobutadene	100
	Hexachloroethane	100
	2-Methylphenol	100
	3-Methylphenol	100
	4-Methylphenol	100
	Nitrobenzene	100
	Pentachlorophenol	100
	Pyridine	100
	2,4,5-Trichlorophenol	100
	2,4,6-Trichlorophenol	100
	Acenaphthene	100
	Acenaphthylene	100
	Anthracene	100
	Benzo(a)anthracene	100
	Benzo(b)fluoranthene	100
	Benzo(k)fluoranthene	100
	Benzo(a)pyrene	100
	Benzo(ghi)perylene	100
	Benzyl butyl phthalate	100
	bis(2-chloroethyl)ether	100
	bis(2-chloroethoxy)methane	100

TABLE 6
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
BNA	bis(2-ethylhexyl)phthalate	100
	bis(2-chloroisopropyl)ether	100
	4-Bromophenyl phenyl ether	100
	2-Chloronaphthalene	100
	4-Chlorophenyl phenyl ether	100
	Chrysene	100
	Dibenzo(a,h)anthracene	100
	Di-n-butylphthalate	100
	1,3-Dichlorobenzene	100
	1,2-Dichlorobenzene	100
	3,3'-Dichlorobenzidine	100
	Diethyl phthalate	100
	Dimethyl phthalate	100
	2,4-Dinitrotoluene	100
	2,6-Dinitrotoluene	100
	Di-n-octylphthalate	100
	Fluoranthene	100
	Fluorene	100
	Hexachlorobenzene	100
	Hexachlorobutadiene	100
	Hexachloroethane	100
	Indeno(1,2,3-cd)pyrene	100
	Isophorone	100
	Naphthalene	100
	Nitrobenzene	100
	n-Nitrosodi-n-propylamine	100
	Phenanthrene	100
	1,2,4-Trichlorobenzene	100
	4-Chloro-3-methylphenol	100
	2-Chlorophenol	100
	2,4-Dichlorophenol	100
	2,4-Dimethylphenol	100
	2,4-Dinitrophenol	100
	2-Methyl-4,6-dinitrophenol	100
	2-Nitrophenol	100
	4-Nitrophenol	100
	Pentachlorophenol	100
	Phenol	100

TABLE 6
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
BNA	2,4,6-Trichlorophenol	100
	Acetophenone	100
	Atrazine	100
	Caprolactum	100
	Benzaldehyde	100
	1,1'-Biphenyl	100
	Safrole	100
	1,4-Dioxane	100
	Pronamide	100
	p-Chlorobenzilate	100
	Phenacetin	100
	Ethyl methanesulfonate	100
	2-Picoline	100
	Phorate	100
	Quinoline	100
	Aniline	100
	Azobenzene	100
	Benzoic Acid	100
	bis-2-EthylhexylAdipate	100
	Carbazole	100
	Dibenzofuran	100
	Hexachlorocyclopentadiene	100
	n-Nitrosodimethylamine	100
	n-Nitrosodiphenylamine	100
	1-Methylnaphthalene	100
	1,2-Dinitroboenzene	100
	1,3-Dinitrobenzene	100
	1,4-initrobenzene	100
	2-Methylnaphthalene	100
	2-Nitroaniline	100
	2,3,4,6-Tetrachlorophenol	100
	2,3,5,6-Tetrachlorophenol	100
	3-Nitronaniline	100
	4-Chloroaniline	100
	4-Methylphenol	100
	4-Nitroaniline	100

TABLE 6
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
Pest TCLP	Heptachlor	0.5
	Heptachlor epoxide	0.5
	Lindane	0.5
	Endrin	0.5
	Methoxychlor	1.0
Pesticides/ NPDES	Alrin	1.0
	Alpha-BHC	1.0
	beta-BHC	1.0
	delta-BHC	1.0
	gamma-BHC (Lindane)	1.0
	4,4'-DDD	1.0
	4,4'-DDE	1.0
	4,4'-DDT	1.0
	Dieldrin	1.0
	alpha-Endosulfan	1.0
	beta-Endosulfan	1.0
	Endosulfan Sulfate	1.0
	Endrin	1.0
	Heptachlor	1.0
	Heptachlor Epoxide	1.0
TPH	Diesel	500 ug/L

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Canton

SOP No. NC-OP-038, Rev. 3
Effective Date: 05/14/13

Title: SEPARATORY FUNNEL EXTRACTION OF ORGANIC COMPOUNDS FROM WATERS BASED ON METHOD SW846 3510C AND 600 SERIES

[Methods: SW846 3510C and 600 Series]

Approvals (Signature/Date):

[redacted]	05/13/13	[redacted]	05/13/13
Technology Specialist	Date	Health & Safety Coordinator	Date
Rebecca Strait	05/10/13	[redacted]	05/10/13
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. NC-OP-038, Rev 2, dated 02/05/13

Facility Distribution No. _____ **Distributed To: _____**

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1. SCOPE AND APPLICATION

1.1 This SOP describes procedures for preparation (extraction) of semivolatile organic analytes in aqueous, and TCLP leachate, matrices for analysis by Gas Chromatography (GC) and Gas Chromatography/ Mass Spectrometry (GC/MS) using Separatory Funnel Extraction. The procedures are based on SW846 and 600 series methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.1.1. Extraction procedures for the following determinative methods are covered: 8081A, 8081B, 8082, 8082A, 8270C, 8270D, 8015B, 8015C, 608, and 625.

1.1.2. The extraction procedures here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

2. SUMMARY OF METHOD

2.1. Separatory Funnel Extraction

2.1.1 A measured volume of sample, (typically one liter, or 250 mL for reduced volume extraction requiring large volume injection) is adjusted, if necessary, to a specified pH and serially extracted with methylene chloride using a separatory funnel.

2.2. Concentration

2.2.1 Procedures are presented for drying and concentration of the extract to final volume for analysis.

3. DEFINITIONS

3.1. Definitions of terms and acronyms used in this SOP may be found in the glossary of the TestAmerica Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.3. The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds must be prepared in the hood.

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3 2 mg/m^3 - Ceiling	This material will cause burns if comes into contact with the skin or eyes. Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.

Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride must be conducted in a fume hood with the sash closed as far as the operations will permit. If more than 500 mL of methylene chloride is spilled, evacuate the area until the area has been cleaned by EH&S.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus and glass separatory funnels have ground glass joints that can become stuck. Technicians must use Kevlar or other cut/puncture-resistant gloves when separating stuck joints.

5.9. 3510 Separatory Funnel

5.9.1. The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Initial venting must be done immediately after the sample container has been sealed and inverted. Periodic venting may be necessary during the extraction. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity. The use of a face shield over safety glasses or goggles is recommended. Keep the sash on the fume hood as low as reasonably possible.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware must be cleaned per Glassware Washing, SOP NC-QA-014.

6.2. Equipment and supplies for extraction procedures:

EQUIPMENT AND SUPPLIES	Sep Fun.	Conc
Separatory funnel: 2 L	√	
Separatory funnel rack	√	
pH indicator paper, ranges: 0-14, 7.5-14, 0-6	√	
Class A Graduated cylinder: 1 liter. (other sizes may be used as needed)	√	√
Centrifuge	√	
Methylene chloride collection tank	√	√
Initial volume template	√	
Solvent dispenser pump or 100 mL Class A graduated cylinder		√
Boiling chips: contaminant-free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent)		√
Beakers: 250 & 400 mL, graduated 450mL wide-mouth glass jars	√ √	√ √
Kuderna-Danish (K-D) apparatus: 500 mL		√
Concentrator tube: 10 mL, attached to K-D with clips		√
Snyder column: three-ball macro		√
Water Bath: heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.		√
Vials: glass, 2 mL, 2.5mL, and 40 mL capacity with Teflon®-lined screw-cap		√
Nitrogen blowdown apparatus		√
Nitrogen: reagent grade.		√
Culture tubes: 10 mL, 16 mmx100 mm		√
Microliter pipette, syringe 1 mL	√	
Glass wool	√	
Glass funnel: 75 X 75 mm	√	√
Disposable pipettes, 5 ¾ in, and 9in.	√	√
Aluminum foil	√	√
Paper towels	√	√

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better unless otherwise specified.

REAGENTS	Sep Fun.	Conc
Sodium hydroxide (NaOH), pellets: reagent grade	√	
Sodium hydroxide solution, 10 N: dissolve 40 g of NaOH in reagent water and dilute to 100 mL.	√	
Sulfuric acid (H_2SO_4), concentrated: reagent grade	√	
Sulfuric acid (1:1): carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.	√	
Hydrochloric acid (HCl)		
Organic free reagent water	√	
Sodium sulfate (Na_2SO_4), granular, anhydrous: purify by heating at 400°C a minimum of two hours.	√	√
Extraction/exchange solvents: methylene chloride, hexane, acetonitrile, acetone, pesticide quality or equivalent	√	√
Acetone, methylene chloride: used for cleaning	√	√
Sodium chloride (NaCl) crystal	√	

7.2. Standards

7.2.1. Stock Standards

7.2.1.1 Stock standards are purchased as certified solutions. Semivolatile stock standards are stored according to manufacturer's instructions. All stock standards must be protected from light. Stock standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampoule is opened if purchased). Standards must be allowed to come to room temperature before use.

7.2.2. Surrogate Spiking Standards

7.2.2.1 Prepare or purchase surrogate spiking standards at the concentrations listed in Table 5. Surrogate spiking standards are purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light, or stored according to manufacturer's instructions. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.3. Matrix Spiking and Laboratory Control Spiking Standards

7.2.3.1 The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Table 6. Spiking standards are purchased or prepared as dilutions of the stock standards. Spiking solutions must be refrigerated and protected from light, or stored according to manufacturer's instructions. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.4 See SOP NC-QA-017 for additional information on Standards and Reagents.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1 The holding time for aqueous samples is seven days from sampling to extraction.

8.3.2 For TCLP leachates, the holding time is 14 days from sampling to the leach process. The extraction holding time seven days from when the TCLP Leach tumbling has been completed, excluding the filtration step, to the extraction step. If the filtration step requires extended times, this time counts as part of the seven-day holding time.

8.3.3. Analysis of the extracts is completed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

9.1.1. The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS, and a matrix spike / matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS / MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD. See Policy QA-003 for further definition of the batch.

9.2. Sample Count

9.2.1. Laboratory-generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count. Field samples are included.

9.3. Method Blank

9.3.1. A method blank consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. See Table 3 for the appropriate amount of surrogate to use for each analytical method. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

9.3.2. Aqueous Method Blanks use 1000 mL of reagent water (250 mL for reduced volume extraction) spiked with the surrogates. The method blank goes through the entire analytical procedure.

9.3.3. TCLP method blanks use 250 mL of leachate fluid spiked with the surrogates. SPLP method blanks use 1000 mL of leachate fluid spiked with the surrogates. The leachate may optionally be diluted to 1000 mL with reagent water. The method blank goes through the entire analytical procedure.

9.4. Laboratory Control Sample (LCS)

9.4.1. Laboratory Control Samples are well-characterized laboratory-generated samples used to monitor the laboratory day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure.

9.4.2. The LCS is made up in the same way as the method blank (see Sections 9.3.1 through 9.3.3), but spiked with the LCS standard and the surrogates. See Tables 3 and 4 for the appropriate amount of spike to use for each analytical method.

9.5. Surrogates

9.5.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.5.2. Each applicable sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. See Table 3 for the appropriate amount of surrogate spike to use for each analytical method.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1. A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and matrix spike. See Tables 3 and 4 for the appropriate amount of spike to use for each analytical method.

9.7. Initial Demonstration of Capability

9.7.1. The initial demonstration and method detection limit studies described in Section 13 must be acceptable before analysis of samples may begin.

9.8 Control Limits

9.8.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.8.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMs.

9.9 Method Detection Limits (MDLs) and MDL Checks

9.9.1 MDLs and MDL Checks are established by the laboratory as described in SOPs CA-Q-S-006 and NC-QA-021.

9.9.2 MDLs are accessible via LIMs.

9.10 Nonconformance and Corrective Action

9.10.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action.

10. CALIBRATION AND STANDARDIZATION

10.1. On a weekly basis, measure the appropriate volume of solvent into the appropriate size glass vial using a gastight syringe that is manufactured to a certified volume delivery tolerance of ± 0.01 mL. The "standard" glass vial is sealed, and the meniscus is noted by marking a line on the bottle. The glass vials containing the sample extracts are then compared against the "standard" glass vial to ensure the final volume is consistently 1.0 ± 0.01 mL. A log is kept of the glass vial lot number and preparation date.

11. PROCEDURE

Refer to SOP NC-QA-016 for information on DoD samples.

11.1. Procedural Variations

11.1.1 Procedural variations are allowed only if deemed necessary in the professional judgment of QA, Operations Supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance memo and approved by a supervisor. The Nonconformance memo will be filed in the project file. Procedural variations are not allowed for Ohio VAP projects.

11.2. Separatory Funnel Liquid/Liquid Extraction of Water Samples

11.2.1. Remove surrogate and matrix spiking solutions from refrigerator and allow to return to room temperature.

11.2.2. Measure the initial sample pH by inserting a disposable pipette into the sample, and placing a drop of sample on the wide-range pH paper. Record in LIMS.

11.2.3. Measure the initial volume using the volume template. Place the template next to the sample bottle and read the volume marking from the template. Record this volume on the benchsheet. The normal sample volume is 1 liter (or 250 mL for reduced volume preps). Other sample volumes may be used to obtain specific reporting limits, and reduced sample volumes, diluted to 1 liter with reagent water, may be used for very dirty samples. If the sample cannot be prepared using a separatory funnel due to matrix issues, a waste dilution may be required. Refer to Section 11.3 for the waste dilution procedure.

11.2.4. Prepare a method blank, LCS, and MS/MSD for each batch as specified in Section 9 of this SOP. Use 1 L of reagent water for method blanks and LCS. Use 500 mL of sample for the MS/MSD. The LCS and MS/MSD are spiked with the surrogate and matrix spike solutions, the method blank only with the surrogates. See Tables 3 and 4 for the appropriate amount of spike solution to use for each analytical method.

11.2.5. Use 250 mL of leachate for TCLP pesticides and TCLP semivolatiles measured in a beaker. Use 1000 mL of leachate for SPLP semivolatiles and SPLP pesticides.

11.2.6. For a TCLP method blank and LCS, measure 250 mL of the buffer solution used in the leaching procedure and transfer to the separatory funnel. Add 60 mL of methylene chloride to the separatory funnel. The TCLP leachate may be diluted to approximately 1 liter before extraction if needed due to matrix. For an SPLP method blank and LCS, measure 1000 mL of the buffer solution using the volume template and transfer to the separatory funnel.

11.2.7. Spike the samples with the appropriate surrogate and/or spike solutions. All samples are spiked in the original sample bottle.

11.2.8. Pour the sample into a separatory funnel. Rinse the sample bottle with methylene chloride. Add the rinsate to the separatory funnel. Add 60 mL of methylene chloride per sample (40mL for reduced volume preps). If the extraction is for reduced volume 8270 analysis, add approximately 5 grams of NaCl crystal. Place a labeled collection jar under each appropriate separatory funnel. Place a small amount of glass wool into a funnel and fill with anhydrous sodium sulfate. Place a funnel containing sodium sulfate on each collection jar.

11.2.9. Adjust sample pH as indicated in Table 1 for th$_e$ initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH, as necessary. Recheck the sample by inserting a disposable pipette into the sample, and placing a drop

of sample onto the pH paper. Record adjusted pH, spiking volumes, and standard numbers on the benchsheet. Return spiking solutions that require cold storage to the refrigerator as soon as possible.

11.2.10. Seal and shake or rotate the separatory funnel vigorously for two minutes with periodic venting to release excess pressure. An autoshaker may be used to shake and rotate the separatory funnel.

Warning: Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting must be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

11.2.11. Allow the organic layer to separate from the water phase until complete visible separation has been achieved. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. If the emulsion cannot be broken (recovery of <80% of the methylene chloride*), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in continuous liquid-liquid extraction (Section 11.2). If this is done, the sample must be extracted as part of a valid CLLE batch.

***Note**: 15 - 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 35 mL from the first shake and still be acceptable. Subsequent shakes must recover at least 50 mL of solvent.

11.2.12. Drain the solvent extract from the separatory funnel through the prepared filtration funnel into a clean glass container. The extract may be drained directly into the KD flask. Close the stopcock just before the water level begins draining out of the separatory funnel. If the sodium sulfate becomes saturated with water, replace the existing sodium sulfate with fresh drying agent.

11.2.13. Repeat the extraction process two more times using fresh 60 mL portions of solvent, combining the three solvent extracts in the collection container.

11.2.14 If extraction at a secondary pH is required, replace the filtration funnel and adjust the pH of the sample in the separatory funnel to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure by inserting a disposable pipette into the sample, and placing a drop of sample onto the pH paper. Record the adjusted pH on the benchsheet. Serially extract with three 60 mL portions of methylene chloride (40 mL for reduced volume preps), as outlined in Steps 11.2.7 to 11.2.10. Collect these three extracts in the same container used for the previous fraction.

11.2.15. Rinse the extract residue from the sodium sulfate by pouring 20-30 mL of clean methylene chloride through the funnel and into the collection container.

11.2.16. Dispose of solvent and water remaining in the extractor into the appropriate waste container.

11.2.17. Cover with aluminum foil and refrigerate if the extract is not concentrated immediately. Refer to Section 11.4 for concentration.

11.3 Concentration

11.3.1 According to the type of sample, different solvents and final volumes will be required. Refer to Table 2 for the appropriate final volumes and concentrations.

11.3.2 Kuderna-Danish (KD) Method

11.3.2.1 Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube to the 500 mL KD flask. Label the CT and KD. Transfer the sample to the labeled K-D flask, filtering samples through funnels filled with sodium sulfate. Rinse the funnel with 20-30 mL of methylene chloride to complete the quantitative transfer.

11.3.1.2 Add one or two clean boiling chips to the KD flask and attach a three-ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column. (This is important to ensure that the balls are not stuck, and the column will work properly). Attach to the KD flask.

11.3.1.3 Place the KD apparatus on a water bath (90-98°C) so the tip of the concentrator tube is submerged. The water level must not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter; but the chambers should not flood.

11.3.1.4 Concentrate to 15-20 mL. If the determinative method requires a solvent exchange, add the appropriate exchange solvent to the top of the Snyder Column, and then continue the water bath concentration back down to 5-8 mL. Refer to Table 2 for details of exchange solvents and final volumes. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.3.2.5 Remove the KD apparatus from the water bath and allow to cool for

a minimum of 10 minutes. If the level of the extract is above the level of the concentrator tube joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

11.4 Nitrogen Evaporation to Final Concentration

11.4.1 Transfer the CT to the evaporation apparatus.

11.4.2 Place the tube in a warm water bath that is at least 5°C below the boiling temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but must not create splattering of the extract.

Boiling points of commonly used solvents are:

Methylene chloride	40°C
Acetone	56°C
Hexane	69°C
Acetonitrile	82°C
Toluene	111°C

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.4.3 Refer to Table 1 to determine the final volume needed for a specific test method. Evaporate to slightly less than the required final volume.

11.4.4. Quantitatively transfer the extract to the appropriate final container and dilute to the appropriate final volume using the "standard" glass vial noted in Section 10.1.
Cap the sample and affix the appropriate label. The sample is now ready for analysis.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

11.5 Analytical Documentation

11.5.1 Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.5.2 Record all standards and reagents in the LIMS Reagents Module. All standards and reagents are assigned a unique number for identification.

11.5.3 Record all sample and associated QC information directly into LIMS. Level I and Level II review is performed in LIMS.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

13.1.1. Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which must contain all the analytes of interest. The spiking level must be equivalent to a mid-level calibration. (For certain tests, more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

13.1.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.1.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs. See SOPs NC-GC-038, NC-MS-018, NC-MS-003, and
NC-GC-007 for detailed information on the determinative methods.

13.2. Training Qualification

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.2.2 Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method

the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. The following waste streams are produced when this method is carried out.

15.2.1. Extracted aqueous samples contaminated with methylene chloride. This tank is then periodically rolled to the tank room, the pH is verified, the contents are neutralized with sodium bicarbonate, the pH re-verified and the Dichloromethane waste drained into a waste drum located outside the building. The wastewater is discharged to the POTW.

15.2.2. Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride/acetone or acetone/hexane from the extract drying step. These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.3. **Assorted flammable solvent waste from various rinses**. These wastes are put into the halogenated/non-halogenated 25-gallon solvent waste container located under the fume hood in extractions.

15.2.4. **Methylene chloride waste from various rinses:** These wastes are disposed of in the liquid-liquid separation unit.

15.2.5. **Hexane-Hexane waste:** These samples are to be disposed in the flammable waste.

15.2.6. **Waste Hexane in vials.** These vials are placed in the vial waste located in the GC prep laboratory.

15.2.7. **Waste Methylene Chloride sample vials**. These vials are placed in the vial waste located in the GC prep laboratory.

15.2.8. Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCBs must be segregated into their own waste stream. PCB wastes are collected in one of three waste streams, solid PCB, liquid PCB, and PCB vial waste.

15.2.9. Solvent Recovery System Waste. Methylene Chloride waste from the Solvent Recovery System is collected and disposed of in the liquid-liquid separation unit. Acetone/Methylene Chloride waste from this system is disposed of in the flammable waste containers located in the laboratory.

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III (December 1996). Sections 3500B, 3510C, and 3580A

16.1.2. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.4. Corporate Quality Management Plan (CQMP), current version

16.1.5 Federal Register - Environmental Protection Agency, 40 CFR, Part 136, Volume 49, No. 209, October 26, 1984, Method 625

16.1.6 EPA 600, Methods for Chemical Analysis of Water and Wastes, Method 608

16.1.7 Revision History

Historical File:		Revision 3.4: 10/16/98		Revision 0: 03/12/08 (NC-OP-032)
(formerly CORP-OP-0001NC)		Revision 3.5: 04/22/99		Revision 1: 01/07/09 (NC-OP-032)
		Revision 3.6: 05/13/99		Revision 0: 03/03/11 (NC-OP-038)
		Revision 3.7: 03/20/01		Revision 1A: 04/24/12
		Revision 3.8: 05/23/01		Revision2: 02/05/13
		Revision 3.9: 04/22/02		
		Revision 4.0: 02/04/03		
		Revision 4.1: 10/07/03		
		Revision 4.2: 01/30/06		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5. Supplemental Practices for DoD Project Work SOP, NC-QA-016

16.2.6. Gas Chromatographic Analysis based on Method 8000B, 8021B, 8081A, 8081B, 8082, 8082A, 8151A, 8015B, 8015C, and 615, NC-GC-038

16.2.7. GC/MS Analysis based on Method 8270C and 8270D, NC-MS-018

16.2.8. Analysis of Pesticides and PCBs by EPA Method 608, NC-GC-007

16.2.9. GC/MS Semivolatile Organic Compounds Capillary Column Technique Based on EPA Method 625, NC-MS-003

16.2.10. Standards and Reagents, NC-QA-017

17. MISCELLANEOUS

17.1. Modifications from Reference method

17.1.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.1.2. Spiking levels for method 608 have been reduced by a factor of ten to bring the levels within the normal calibration range of the instrument.

17.2. Tables

TABLE 1 Liquid /Liquid Extraction Conditions		
Determinative Method	**Initial Ext. pH**	**Secondary Ext. pH**
BNA	Acid ext; 1-2 or Base ext; 11-12	Acid ext; 1-2 or Base ext; 11-12
Pesticide/PCB	5-9	None
TPH	As received	None

Note: If the laboratory has validated acid only 8270 extraction for the target compound list required, then the base extraction step may be omitted. The required validation consists of a four-replicate initial demonstration of capability and a method detection limit study (see Section 13). Additionally, either of the base or acid fractions of Method 8270 can be run first.

TABLE 2 Final Volumes and Exchange Solvents		
Type	**Exchange Solvent for Analysis**	**Final Volume for Analysis in mL**
BNA	N/A	2.0 mL
PCB	Approximately 18 mL Hexane – water	5.0 for H2O 2.0 for H2O*
Pesticides	Approximately 18 mL Hexane	5.0 for H2O 2.0 for H2O
Pesticides/TCLP	Approximately 18 mL Hexane	3.0 mL
BNA – SIM	N/A	2.0 mL 5.0 mL for reduced volume preps
TPH	N/A	5.0 mL

* Michigan work requires a final volume of 2 mL.

Note: Different final volumes may be necessary to meet special client reporting limit requirements.

TABLE 3 Surrogate Spiking Solutions		
Analyte Group	**Surrogate Spike Solution ID**	**Volume (mL)**
BNA	100/150 ppm BNA	0.2
BNA – SIM	100/150 ppm BNA	0.2 / 0.02
Pesticides	0.2 ppm DCB/TCX	1.0
TPH	40ng Nonane (C9)	1.0
PCB	0.2 ppm DCB/TCX	1.0

* Note surrogate spiking levels are adjusted for reduced volume preps which utilize large volume injection

TABLE 4 Matrix Spike and LCS Solutions		
Analyte Group	**Matrix Spike Solution ID**	**Volume (mL)**
BNA	100 ppm BNA All-Analyte Spike and Restek Spike	0.2
BNA – SIM	100 ppm BNA All-Analyte Spike and Restek Spike	0.2 / 0.02
Pesticides	Pest NPDES Spike	1.0
Pesticides TCLP	Pest TCLP Spike	1.0
PCB	10 ppm PCB Spike	1.0
TPH	See Spike List – Table 6	1.0

* Note surrogate spiking levels are adjusted for reduced volume preps which utilize large volume injection

TABLE 5
Surrogate Spike Components

Analyte Group	Compounds	Conc. (µg/mL)
BNA	2-Fluorobiphenyl	100
	Nitrobenzene-d5	100
	p-Terphenyl-d14	100
	2-Fluorophenol	150
	Phenol-d6	150
	2,4,6-Tribromophenol	150
	1,2-Dichlorobenzene-d4	100
	2-Chlorophenol-d4	150
Pesticides PCB	Decachlorobiphenyl	0.2
	Tetrachloro-m-xylene	0.2
TPH	Nonane (C9)	40.0

TABLE 6
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
BNA	Acenaphthene	100
	4-Chloro-3-Methylphenol	100
	2-Chlorophenol	100
	1,4-Dichlorobenzene	100
	2,4-Dinitrotoluene	100
	4-Nitrophenol	100
	Pentachlorophenol	100
	Phenol	100
	Pyrene	100
	1,2,4-Trichlorobenzene	100
	1,4-Dichlorobenzene	100
	2,4-Dinitrotoluene	100
	Hexachlorobenzene	100
	Hexachlorobutadiene	100

TABLE 6
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
BNA	Hexachloroethane	100
	2-Methylphenol	100
	3-Methylphenol	100
	4-Methylphenol	100
	Nitrobenzene	100
	Pentachlorophenol	100
	Pyridine	100
	2,4,5-Trichlorophenol	100
	2,4,6-Trichlorophenol	100
	Acenaphthene	100
	Acenaphthylene	100
	Anthracene	100
	Benzo(a)anthracene	100
	Benzo(b)fluoranthene	100
	Benzo(k)fluoranthene	100
	Benzo(a)pyrene	100
	Benzo(ghi)perylene	100
	Benzyl butyl phthalate	100
	bis(2-chloroethyl)ether	100
	bis(2-chloroethoxy)methane	100
	bis(2-ethylhexyl)phthalate	100
	bis(2-chloroisopropyl)ether	100
	4-Bromophenyl phenyl ether	100
	2-Chloronaphthalene	100
	4-Chlorophenyl phenyl ether	100
	Chrysene	100
	Dibenzo(a,h)anthracene	100
	Di-n-butylphthalate	100
	1,3-Dichlorobenzene	100
	1,2-Dichlorobenzene	100
	3,3'-Dichlorobenzidine	100
	Diethyl phthalate	100
	Dimethyl phthalate	100
	2,4-Dinitrotoluene	100
	2,6-Dinitrotoluene	100
	Di-n-octylphthalate	100
	Fluoranthene	100
	Fluorene	100
	Hexachlorobenzene	100

TABLE 6
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
BNA	Hexachlorobutadiene	100
	Hexachloroethane	100
	Indeno(1,2,3-cd)pyrene	100
	Isophorone	100
	Naphthalene	100
	Nitrobenzene	100
	n-Nitrosodi-n-propylamine	100
	Phenanthrene	100
	1,2,4-Trichlorobenzene	100
	4-Chloro-3-methylphenol	100
	2-Chlorophenol	100
	2,4-Dichlorophenol	100
	2,4-Dimethylphenol	100
	2,4-Dinitrophenol	100
	2-Methyl-4,6-dinitrophenol	100
	2-Nitrophenol	100
	4-Nitrophenol	100
	Pentachlorophenol	100
	Phenol	100
	2,4,6-Trichlorophenol	100
	Acetophenone	100
	Atrazine	100
	Caprolactum	100
	Benzaldehyde	100
	1,1'-Biphenyl	100
	Safrole	100
	1,4-Dioxane	100
	Pronamide	100
	p-Chlorobenzilate	100
	Phenacetin	100
	Ethyl methanesulfonate	100
	2-Picoline	100
	Phorate	100
	Quinoline	100
	Aniline	100
	Azobenzene	100
	Benzoic Acid	100
	bis-2-Ethylhexyl Adipate	100
	Carbazole	100

TABLE 6
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
	Dibenzofuran	100
	Hexachlorocyclopentadiene	100
	n-Nitrosodimethylamine	100
	n-Nitrosodiphenylamine	100
	1-Methylnaphthalene	100
	1,2-Dinitroboenzene	100
	1,3-Dinitrobenzene	100
	1,4-initrobenzene	100
	2-Methylnaphthalene	100
	2-Nitroaniline	100
	2,3,4,6-Tetrachlorophenol	100
	2,3,5,6-Tetrachlorophenol	100
	3-Nitronaniline	100
	4-Chloroaniline	100
	4-Methylphenol	100
	4-Nitroaniline	100
Pesticides TCLP	Heptachlor	0.5
	Heptachlor epoxide	0.5
	Lindane	0.5
	Endrin	0.5
	Methoxychlor	1.0
Pesticides NPDES	Aldrin	1.0
	Alpha-BHC	1.0
	beta-BHC	1.0
	delta-BHC	1.0
	gamma-BHC (Lindane)	1.0
	4,4'-DDD	1.0
	4,4'-DDE	1.0
	4,4'-DDT	1.0
	Dieldrin	1.0
	alpha-Endosulfan	1.0
	beta-Endosulfan	1.0
	Endosulfan Sulfate	1.0
	Endrin	1.0
	Heptachlor	1.0
	Heptachlor Epoxide	1.0
TPH	Diesel Fuel	500 µg/L

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica North Canton

SOP No. NC-OP-039, Rev. 0
Effective Date: 03/03/11

Title: SONICATION EXTRACTION OF ORGANIC COMPOUNDS FROM SOILS BASED ON METHOD SW846 3550C AND WASTE DILUTION BASED ON METHOD 3580A

[Methods: SW846 3550C and 3580A]

Approvals (Signature/Date):

Technology Specialist	02/22/11 Date	Health & Safety Coordinator	02/18/11 Date
Quality Assurance Manager	03/02/11 Date	Laboratory Director	02/27/11 Date
Technical Director	03/03/11 Date		

This SOP was previously identified as SOP No. NC-OP-032, Rev 1, dated 01/07/09, which is now split into four individual SOPs

Facility Distribution No. _____ **Distributed To: _____**

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List of Tables

1. SCOPE AND APPLICATION

1.1 This SOP describes procedures for preparation (extraction) of semivolatile organic analytes in soil matrices for analysis by Gas Chromatography (GC) and Gas Chromatography/ Mass Spectrometry (GC/MS) using Sonication Extraction. The procedures are based on SW846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.1.1. Extraction procedures for the following determinative methods are covered: 8081A, 8081B, 8082, 8082A, 8270C, 8270D, 8015B, 8015C, 608, and 625.

1.1.2. The extraction procedures here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

2. SUMMARY OF METHOD

2.1. Sonication Extraction

A measured weight of sample, typically 30 g, is mixed with anhydrous sodium sulfate until free flowing. This is solvent extracted three times using an ultrasonic horn.

2.2. Concentration

Procedures are presented for drying and concentration of the extract to final volume for analysis.

3. DEFINITIONS

3.1. Definitions of terms and acronyms used in this SOP may be found in the glossary of the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated must be removed and discarded, other gloves must be cleaned immediately.

5.3. The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds must be prepared in the hood.

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride must be conducted in a fume hood with the sash closed as far as the operations will permit. If more than 500 mL of methylene chloride is spilled, evacuate the area until the area has been cleaned by EH&S.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8. During Kuderna-Danish(KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus and glass separatory funnels have ground glass joints which can become stuck. Technicians must use Kevlar or other cut/puncture-resistant gloves when separating stuck joints.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware must be cleaned per Glassware Washing, SOP NC-QA-014.

6.2. Equipment and supplies for extraction procedures:

EQUIPMENT AND SUPPLIES	Soni	Conc
Solvent dispenser pump or 100 mL graduated cylinder		√
Boiling chips: contaminant-free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent)		√
450mL wide-mouth glass jars	√	√
Balance: >100 g capacity, accurate ± 0.1 g	√	√
Sonicator (at least 300 watts)	√	
Sonicator horn, 3/4 inch	√	
Micro-tip sonicator horn	√	
Kuderna-Danish (K-D) Apparatus: 500 mL		√
Concentrator tube: 10 mL, attached to K-D with clips		√

EQUIPMENT AND SUPPLIES	Soni	Conc
Snyder column: three-ball macro		√
Water bath: heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.		√
Vials: glass, 2 mL, and 40 mL capacity with Teflon®-lined screw-cap		√
Nitrogen blowdown apparatus		√
Nitrogen: reagent grade		√
Culture tubes: 10 mL, 16 mmx100 mm		√
Microliter pipette , syringe 1 mL	√	
Glass wool	√	√
Glass funnel: 75 X 75 mm	√	√
Disposable pipettes, 5 ¾ in, and 9in.	√	√
Aluminum foil	√	√
Paper towels	√	√

7 REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better unless otherwise specified.

REAGENTS	Soni	Conc
Hydrochloric acid (HCl)	√	√
Sodium sulfate (Na_2SO_4), granular, anhydrous: Purify by heating at 400°C a minimum of two hours.	√	√
Magnesium sulfate	√	
Extraction/exchange solvents: Methylene chloride, hexane, acetone, pesticide quality or equivalent	√	√
Acetone, methylene chloride: used for cleaning	√	√

7.2. Standards

7.2.1. Stock Standards

Stock standards are purchased as certified solutions. Semivolatile stock standards are stored at $\leq 6^{o}C$. All stock standards must be protected from light. Stock standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampoule is opened if purchased). Standards must be allowed to come to room temperature before use.

7.2.2. Surrogate Spiking Standards

Prepare or purchase surrogate spiking standards at the concentrations listed in Table 4. Surrogate spiking standards are purchased or prepared as dilutions of the stock

standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.3. Matrix Spiking and Laboratory Control Spiking Standards

The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Table 5. Spiking standards are purchased or prepared as dilutions of the stock standards.

Spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.4 See SOP NC-QA-017 for additional information on Standards and Reagents.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps. Sample containers other glass, must be documented in a Non-Conformance Memo.

8.3. Holding Times

8.3.1 The holding time for solid and waste samples is 14 days from sampling to extraction.

8.3.2 Analysis of the extracts is completed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

9.1.1. The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS, and a matrix spike / matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS / MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD. See Policy QA-003 for further definition of the batch.

9.2. Sample Count

9.2.1. Laboratory-generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count. Field samples are included.

9.3. Method Blank

9.3.1. A method blank consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. See Table 2 for the appropriate amount of surrogate to use for each analytical method. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

9.3.2. Solid method blanks use approximately 30 g of sodium sulfate spiked with the surrogates. See 2 for the appropriate amount of surrogate to use for each analytical method. The method blank goes through the entire analytical procedure.

9.4. Laboratory Control Sample (LCS)

9.4.1. Laboratory Control Samples are well-characterized laboratory-generated samples used to monitor the laboratory day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure.

9.4.2. The LCS is made up in the same way as the method blank (see Sections 9.3.1 through 9.3.2), but spiked with the LCS standard and the surrogates. See Tables 2 and 3 for the appropriate amount of spike to use for each analytical method.

9.5. Surrogates

9.5.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.5.2. Each applicable sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. See Table 2 for the appropriate amount of surrogate spike to use for each analytical method.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1. A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and matrix spike.

See Tables 2 and 3 for the appropriate amount of spike to use for each analytical method.

9.7. Initial Demonstration of Capability

9.7.1. The initial demonstration and method detection limit studies described in Section 13 must be acceptable before analysis of samples may begin.

9.8 Control Limits

9.8.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.8.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMs (QC Browser program).

9.9 Method Detection Limits (MDLs) and MDL Checks

9.9.1 MDLs and MDL Checks are established by the laboratory as described in SOPs CA-Q-S-006 and NC-QA-021.

9.9.2 MDLs are accessible via LIMs (QC Browser program).

9.10 Nonconformance and Corrective Action

9.10.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. On a weekly basis, measure the appropriate volume of solvent into the appropriate size glass vial using a gastight syringe that is manufactured to a certified volume delivery tolerance of ± 0.01 mL. The "standard" glass vial is sealed, and the meniscus is marked by etching a line on the bottle. The glass vials containing the sample extracts are then compared against the "standard" glass vial to ensure the final volume is consistently 1.0 ± 0.01 mL. A log is kept of the glass vial lot number and preparation date.

11. PROCEDURE

Refer to SOP NC-QA-016 for information on DoD samples.

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, chemistry, sample size, or other

parameters. Any variation in procedure must be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC Manager. The Nonconformance memo will be filed in the project file. Procedural variations are not allowed for Ohio VAP projects.

11.2 Sonication

11.2.1 Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

11.2.2 Decant any water layer on a sediment/soil sample into the sample lid or mason jar and return it into the sample after removing the amount needed for the extraction, unless there are specific instructions not to decant the water. Record and document if a water layer was present on the benchsheet. Homogenize the sample by mixing it thoroughly in the container. If this is not possible, place the sample in clean beaker and homogenize. Upon completion of homogenization in the beaker, return sample to original container. Discard foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials, consult with the client (via the Project Manager or Administrator). If the sample cannot be prepared using sonication due to matrix issues, a waste dilution may be required. Refer to Section 11.4 for the waste dilution procedure.

11.2.3 Weigh 30 g of sample ± 0.5 g into a 250 or 400 mL beaker or wide-mouth jar. Record the weight to the nearest 0.01 g in the appropriate column on the benchsheet.

11.2.4 Mix the weighed sample with a spatula adding enough anhydrous sodium sulfate (approximately 30 g) to be free flowing. (If the sample is not free flowing, extraction efficiency may be reduced.) Add the appropriate spike and surrogate solution.

11.2.5 Add appropriate volume of matrix spiking solution to any matrix spike / matrix spike duplicate samples (see Table 3). Add the appropriate volume of the surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes (see Table 2 for appropriate amounts). Refer to Table 5 for details of the surrogate spiking solutions. Add the appropriate matrix spiking solution to each Matrix Spike/Matrix Spike Duplicate (MS/MSD) and LCS. Refer to Tables 2 and 4 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

11.2.6 Prepare a method blank, LCS, and MS/MSD for each batch as specified in Section 9 of this SOP. Use 30 g of sodium sulfate for the method blank and LCS. Use 30 g ± 0.5 g each of parent sample for the MS and MSD samples.

11.2.7 Immediately add approximately 100 mL of solvent to the beaker.

Solvents:

Semivolatile GC/MS, TPH, Organochlorinepesticides and PCBs	1:1 v/v Methylene Chloride / Acetone
8270 (MS) Concrete	Methylene Chloride

Note: Steps 11.2.5 through 11.2.9 must be performed rapidly to avoid loss of the more volatile extractables.

11.2.8 Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.2.9 Sonicate for three minutes, making sure the entire sample is agitated.

Note: Do ***not*** use ***Micro-tip*** probe.

11.2.10 Loosely plug the stem of a 75 mm x 75 mm glass funnel with glass wool. Add 10-20 g of anhydrous sodium sulfate to the funnel cup.

11.2.11 Place the prepared funnel on a labeled collection apparatus (beaker or K-D apparatus).

11.2.12 Decant and filter extracts through the prepared funnel into the collection apparatus (a clean beaker or K-D Apparatus).

11.2.13 Repeat the extraction two more times with approximately 100 mL portions of solvent each time. Decant off extraction solvent after each sonication. On the final sonication, pour the entire sample (sediment and solvent) into the funnel and rinse with an additional 10 mL-20 mL of the appropriate solvent (refer to table in Section 11.2.7).

Note: Alternatively, the three extracts may be collected together and then filtered through the sodium sulfate.

11.2.14 Cover with aluminum foil and refrigerate if the extract is not concentrated immediately. Refer to Section 11.5 for concentration.

11.2.15 Sonicator Tuning: Tune the sonicator according to manufacturer's instructions. The sonicator must be tuned quarterly and if a new horn is installed.

11.3 Medium Level Sonication

11.3.1 Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

11.3.2 Decant any water layer on a sediment/soil sample into the sample lid or mason jar and return it into the sample after removing the amount needed for the extraction, unless there are specific instructions not to decant the water. Record and document if a water layer was present on the benchsheet. Homogenize the sample by mixing it thoroughly in the container. If this is not possible, place the sample in clean beaker and homogenize. Upon completion of homogenization in the beaker, return sample to original container. Discard foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials, consult with the client (via the Project Manager or Administrator).

11.3.3 Weigh 5 g of sample ± 0.1 g into a 40 mL vial. Record the weight to the nearest 0.01 g in the appropriate column on the benchsheet.

11.3.4 Mix the weighed and spiked/surrogated sample with a spatula adding enough anhydrous sodium sulfate to be free flowing.

11.3.5 Add appropriate volume of matrix spiking solution to any matrix spike / matrix spike duplicate samples (see Table 3). Add the appropriate volume of the surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes (see Table 2 for appropriate amounts). Refer to Table 5 for details of the surrogate spiking solutions. Add the appropriate matrix spiking solution to each Matrix Spike/Matrix Spike Duplicate (MS/MSD) and LCS. Refer to Tables 2 and 4 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

11.3.6 Prepare a method blank, LCS, and MS/MSD for each batch as specified in Section 9 of this SOP. Use 5 g of sodium sulfate for the method blank and LCS. Use 5 g ± 0.1 g each of parent sample for the MS and MSD samples.

11.3.7 Immediately add the appropriate mLs of solvent to the vial.

LCS, MS/MSD	18 mL Hexane
Sample and blank	19 mL Hexane

Note: Steps 11.3.5 - 11.3.9 must be performed rapidly to avoid loss of the more volatile extractables.

11.3.8 Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.3.9 Sonicate for two minutes continuous disruption, making sure the entire sample is agitated.

Note: Use ***Micro-tip*** probe.

11.3.10 The samples are now ready for analysis.

11.4 Waste Dilution

11.4.1 Remove surrogate and matrix spiking solutions from refrigerator and allow to return to room temperature.

11.4.2 Label the vial with the sample number. Tare the vial, then transfer approximately 1g of sample to the vial. Record the weight to the nearest ± 0.01g.

11.4.3 For the blank and LCS, add a small amount of the appropriate solvent to the vial. Add appropriate volume of surrogate and spike solutions (Table 2).

11.4.4 Dilute to 10 mL with the appropriate solvent (Methylene Chloride for GC/MS Semi and GCS TPH). Add 10 mL of appropriate solvent (Hexane) for GCS pesticide and/or PCB analysis. Method 8015B waste dilutions are diluted to approximately 10 mL with DCM and are placed on the nitrogen evaporation unit to reduce to a 2 mL final volume.

11.4.5 Cap and shake or vortex each extract.

11.4.6 The sample is now ready for analysis.

11.5 Concentration

According to the type of sample, different solvents and final volumes will be required. Refer to Table 1 for the appropriate final volumes and concentrations.

11.5.1 Kuderna-Danish (KD) Method:

11.5.1.1 Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube to the 500 mL KD flask. Label the CT and KD. Transfer the sample to the labeled K-D flask, filtering samples through funnels filled with sodium sulfate. Rinse the funnel with 20-30 mL of methylene chloride to complete the quantitative transfer.

11.5.1.2 Add one or two clean boiling chips and the extract to be concentrated to the KD flask and attach a three-ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column. (This is important to ensure that the balls are not stuck, and the column will work properly). Attach to the KD flask.

11.5.1.3 Place the KD apparatus on a water bath [90 (for MS Semi samples)-98°C] so the tip of the concentrator tube is submerged. The water level must not reach the joint between the concentrator and the KD flask. At the proper

rate of distillation, the balls will actively chatter; but the chambers should not flood.

11.5.1.4 Concentrate to 15-20 mL. If the determinative method requires a solvent exchange, add the appropriate exchange solvent to the top of the Snyder Column, and then continue the water bath concentration back down to 5-8 mL. Refer to Table 1 for details of exchange solvents and final volumes. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.5.1.5 Remove the KD apparatus from the water bath and allow to cool for a minimum of 10 minutes. If the level of the extract is above the level of the concentrator tube joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

11.6 Nitrogen Evaporation to Final Concentration

11.6.1 Transfer the CT to the evaporation apparatus.

11.6.2 Place the tube in a warm water bath that is at least 5°C below the boiling temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but must not create splattering of the extract.

Boiling points of commonly used solvents are:

Methylene chloride	40°C
Acetone	56°C
Hexane	69°C
Acetonitrile	82°C
Toluene	111°C

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.6.3 Refer to Table 1 to determine the final volume needed for a specific test method. Evaporate to slightly less than the required final volume.

Quantitatively transfer the extract to the appropriate final container and dilute to the appropriate final volume using the "standard" glass vial noted in Section 10.1. Cap the sample and affix the appropriate label. The sample is now ready for analysis.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

11.7 Analytical Documentation

11.7.1 Record all analytical information in the analytical logbook/logsheet which may be in an electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.7.2 All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.7.3 Sample information and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

13.1.1. Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which must contain all the analytes of interest. The spiking level must be equivalent to a mid-level calibration. (For certain tests, more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

13.1.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.1.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs. See SOPs NC-GC-038, NC-MS-018, NC-MS-003, and NC-GC-007 for detailed information on the determinative methods.

13.2. Training Qualification

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. The following waste streams are produced when this method is carried out.

15.2.1. Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride/acetone or acetone/hexane from the extract drying step. These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.2. **Assorted flammable solvent waste from various rinses**. These wastes are put into the halogenated/non-halogenated 25 gallon solvent waste container located under the fume hood in extractions.

15.2.3. **Methylene chloride waste from various rinses:** These wastes are disposed of in the liquid-liquid separation unit.

15.2.4. **Hexane-Hexane waste:** These samples are to be disposed in the flammable waste.

15.2.5. **Waste Hexane in vials.** These vials are placed in the vial waste located in the GC prep laboratory.

15.2.6. **Waste Methylene Chloride sample vials**. These vials are placed in the vial waste located in the GC prep laboratory.

15.2.7. **Extracted solid samples contaminated with methylene chloride/acetone or acetone/hexane.** These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.8. Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCBs must be segregated into their own waste stream. PCB wastes are collected in one of three waste streams, solid PCB, liquid PCB, and PCB vial waste.

15.2.9. Solvent Recovery System Waste. Methylene Chloride waste from the Solvent Recovery System is collected and disposed of in the liquid-liquid separation unit. Acetone/MethyleneChloride waste from this system is disposed of in the flammable waste containers located in the laboratory.

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III (December 1996). Sections 3500B, 3550C and 3580A.

16.1.2. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.4. Corporate Quality Management Plan (CQMP), current version

16.1.5. Federal Register – Environmental Protection Agency, 40 CFR, Part 136, Volume 49, No. 209, October 26, 1984, Method 625

16.1.6. EPA 600, Methods for Chemical Analysis of Water and Wastes, Method 608

16.1.7. Revision History

Historical File:		Revision 3.4: 10/16/98		Revision 0: 03/12/08 (NC-OP-032)
(formerly CORP-OP-0001NC		Revision 3.5: 04/22/99		Revision 1: 01/07/09 (NC-OP-032)
		Revision 3.6: 05/13/99		
		Revision 3.7: 03/20/01		
		Revision 3.8: 05/23/01		
		Revision 3.9: 04/22/02		
		Revision 4.0: 02/04/03		
		Revision 4.1: 10/07/03		
		Revision 4.2: 01/30/06		

16.2 Associated SOPs and Policies, current version

16.2.1 QA Policy, QA-003

16.2.2 Glassware Washing, NC-QA-014

16.2.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4 Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5 Supplemental Practices for DoD Project Work SOP, NC-QA-016

16.2.6 Gas Chromatographic Analysis based on Method 8000B, 8021B, 8081A, 8081B, 8082, 8082A, 8151A, 8015B, 8015C and 615, NC-GC-038

16.2.7 GC/MS Analysis based on Method 8270C and 8270D, NC-MS-018

16.2.8 Analysis of Pesticides and PCBs by EPA Method 608, NC-GC-007

16.2.9 GC/MS Semivolatile Organic Compounds Capillary Column Technique Based on EPA Method 625, NC-MS-003

16.2.10 Standards and Reagents, NC-QA-017

17. MISCELLANEOUS

17.1. Modifications from Reference method

17.1.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.2. Tables

TABLE 1

Final Volumes and Exchange Solvents

Type	Exchange Solvent for Analysis	Final Volume for Analysis in mL
Semivolatiles	N/A	2.0 mL
PCB	Approximately36 mL Hexane - solid	10.0
Pesticides	Approximately18 mL Hexane	10.0
BNA – SIM	N/A	2.0
TPH	N/A	5.0

Note: Different final volumes may be necessary to meet special client reporting limit requirements.

TABLE 2

Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume (mL)
BNA	100/150 ppm BNA	0.2
BNA / SIM	100/150 ppm BNA	0.2 / 0.02
BNA Waste Dilution	100/150 ppm BNA	0.5
PEST	0.2 ppm DCB/TCX	1.0
TPH	40ng Nonane (C9)	1.0
PCB	0.2 ppm DCB/TCX	1.0

TABLE 3
Matrix Spike and LCS Solutions

Analyte Group	Matrix Spike Solution ID	Volume (mL)
BNA	100 ppm BNA All-Analyte Spike and Restek Spike	0.2
	Waste Dilution	0.5
BNA / SIM	100 ppm BNA All-Analyte Spike and Restek Spike	0.2 / 0.02
PEST	Pest NPDES Spike	1.0
PCB	10 ppm PCB Spike	1.0
TPH	See Spike List – Table 5	1.0

TABLE 4
Surrogate Spike Components

Analyte Group	Compounds	Conc. (µg/mL)
BNA	2-Fluorobiphenyl	100
	Nitrobenzene-d5	100
	p-Terphenyl-d14	100
	2-Fluorophenol	150
	Phenol-d6	150
	2,4,6-Tribromophenol	150
	1,2-Dichlorobenzene-d4	100
	2-Chlorophenol-d4	150
PEST	Decachlorobiphenyl	0.2
PCB	Tetrachloro-m-xylene	0.2
TPH	Nonane (C9)	40.0

TABLE 5
LCS and Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
BNA	Acenaphthene	100
	4-Chloro-3-Methylphenol	150
	2-Chlorophenol	150
	1,4-Dichlorobenzene	100
	2,4-Dinitrotoluene	100
	4-Nitrophenol	150
	N-Nitroso-Di-n-Propylamine	100
	Pentachlorophenol	150
	Phenol	150
	Pyrene	100
	1,2,4-Trichlorobenzene	100
	1,4-Dichlorobenzene	100
	2,4-Dinitrotoluene	100
	Hexachlorobenzene	100
	Hexachlorobutadiene	100
	Hexachloroethane	100
	2-Methylphenol	100
	3-Methylphenol	100
	4-Methylphenol	100
	Nitrobenzene	100
	Pentachlorophenol	100
	Pyridine	100
	2,4,5-Trichlorophenol	100
	2,4,6-Trichlorophenol	100
	Acenaphthene	100
	Acenaphthylene	100
	Anthracene	100
	Benzo(a)anthracene	100
	Benzo(b)fluoranthene	100
	Benzo(k)fluoranthene	100
	Benzo(a)pyrene	100
	Benzo(ghi)perylene	100

Type	Compounds	Conc. (µg/mL)
BNA	Benzyl butyl phthalate	100
	Bis(2-chloroethyl)ether	100
	Bis(2-chloroethoxy)methane	100
	Bis(2-ethylhexyl)phthalate	100
	Bis(2-chloroisopropyl)ether	100
	4-Bromophenyl phenyl ether	100
	2-Chloronaphthalene	100
	4-Chlorophenyl phenyl ether	100
	Chrysene	100
	Dibenzo(a,h)anthracene	100
	Di-n-butylphthalate	100
	1,3-Dichlorobenzene	100
	1,2-Dichlorobenzene	100
	1,4-Dichlorobenzene	100
	3,3'-Dichlorobenzidine	100
	Diethyl phthalate	100
	Dimethyl phthalate	100
	2,4-Dinitrotoluene	100
	2,6-Dinitrotoluene	100
	Di-n-octylphthalate	100
	Fluoranthene	100
	Fluorene	100
	Hexachlorobenzene	100
	Hexachlorobutadiene	100
	Hexachloroethane	100
	Indeno(1,2,3-cd)pyrene	100
	Isophorone	100
	Naphthalene	100
	Nitrobenzene	100
	N-Nitrosodi-n-propylamine	100
	Phenanthrene	100
	Pyrene	100
	1,2,4-Trichlorobenzene	100
	4-Chloro-3-methylphenol	100
	2-Chlorophenol	100

Type	Compounds	Conc. (µg/mL)
BNA	2,4-Dichlorophenol	100
	2,4-Dimethylphenol	100
	2,4-Dinitrophenol	100
	2-Methyl-4,6-dinitrophenol	100
	2-Nitrophenol	100
	4-Nitrophenol	100
	Pentachlorophenol	100
	Phenol	100
	2,4,6-Trichlorophenol	100
	Acetophenone	100
	Atrazine	100
	Caprolactum	100
	Benzaldehyde	100
	1,1′-Biphenyl	100
	Safrole	100
	1,4-Dioxane	100
	Pronamide	100
	p-Chlorobenzilate	100
	Phenacetin	100
	Ethyl methanesulfonate	100
	2-Picoline	100
	Phorate	100
	Quinoline	100

Type	Compounds	Conc. (µg/mL)
Pest NPDES/Pest	Alrin	1.0
	Alpha-BHC	1.0
	beta-BHC	1.0
	delta-BHC	1.0
	gamma-BHC (Lindane)	1.0
	4,4'-DDD	1.0
	4,4'-DDE	1.0
	4,4'-DDT	1.0
	Dieldrin	1.0
	alpha-Endosulfan	1.0
	beta-Endosulfan	1.0
	Endosulfan Sulfate	1.0
	Endrin	1.0
	Heptachlor	1.0
	Heptachlor Epoxide	1.0

Diesel Range Organics (8015B) (8015C)Spike	
Compound	**Final Concentration**
Diesel Fuel	500 ug/L

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Canton

SOP No. NC-OP-040, Rev. 2
Effective Date: 08/28/13

Title: SOXHLET (TRADITIONAL) EXTRACTION OF ORGANIC COMPOUNDS FROM SOILS BASED ON METHOD SW846 3540C

[Method: SW846 3540C]

Approvals (Signature/Date):

Technology Specialist	08/20/13 Date	Health & Safety Coordinator	08/21/13 Date
Quality Assurance Manager	08/28/13 Date	Laboratory Director	08/24/13 Date

This SOP was previously identified as SOP No. NC-OP-040, Rev 1-A, dated 04/24/12

Facility Distribution No. _____ **Distributed To: _____**

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1. SCOPE AND APPLICATION

1.1 This SOP describes procedures for preparation (extraction) of semivolatile organic analytes in soil matrices for analysis by Gas Chromatography (GC) and Gas Chromatography/ Mass Spectrometry (GC/MS) using Soxhlet Extraction. The procedures are based on SW846 series methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.1.1. Extraction procedures for the following determinative methods are covered: 8081A, 8081B, 8082, 8082A, 8270C, and 8015B.

1.1.2. The extraction procedures here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

2. SUMMARY OF METHOD

2.1. Soxhlet Extraction (Accelerated and Traditional)

2.1.1 A 30 g sample is mixed with anhydrous sodium sulfate until free flowing. This is extracted with refluxing solvent.

2.2. Concentration

2.2.1 Procedures are presented for drying and concentration of the extract to final volume for analysis.

3. DEFINITIONS

3.1. Definitions of terms and acronyms used in this SOP may be found in the glossary of the TestAmerica Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.3. The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds must be prepared in the hood.

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.

Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride must be conducted in a fume hood with the sash closed as far as the operations will permit. If more than 500 mL of methylene chloride is spilled, evacuate the area until the area has been cleaned by EH&S.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus and glass separatory funnels have ground glass joints that can become stuck. Technicians must use Kevlar or other cut/puncture-resistant gloves when separating stuck joints.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware must be cleaned per Glassware Washing, SOP NC-QA-014.

6.2. Equipment and supplies for extraction procedures:

EQUIPMENT AND SUPPLIES	Sox	Conc
Graduated cylinder: 1 liter. (other sizes may be used as needed)		√
Erlenmeyer flask: 250 mL (other sizes optional)		√
Solvent dispenser pump or 100 mL graduated cylinder	√	√
Round or flat bottom: 250 mL	√	
Boiling chips: contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent)	√	√
Cooling condensers	√	
Heating mantle: rheostat controlled	√	
Auto-timer for heating mantle	√	
Soxgriddle	√	
Beakers: 450mL wide-mouth glass jars		√
Balance: >100 g capacity, accurate ??? g	√	√
Soxhlet extractor	√	
Cellulose and glass thimbles Accelerated soxhlet extractor (Soxtherm™)	√ √	
Kuderna-Danish (K-D) apparatus: 500 mL		√
Concentrator tube: 10 mL, attached to K-D with clips		√
Snyder column: three-ball macro		√
Water bath: heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.		√
Vials: glass, 2 mL and 40 mL capacity with Teflon® lined screw-cap		√
Nitrogen blowdown apparatus		√
Nitrogen: reagent grade.		√
Culture Tubes: 10 mL, 16 mmx100 mm		√
Microliter pipette, syringe 1 mL	√	
Glass wool	√	
Glass funnel: 75 X 75 mm	√	√
Disposable pipettes, 5 ¾ in, and 9in.	√	√
Aluminum foil	√	√
Paper towels	√	√

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better, unless otherwise specified.

REAGENTS	Sox	Conc
Sodium sulfate (Na_2SO_4), Granular, Anhydrous: Purify by heating at 400°C a minimum of two hours.	√	√
Magnesium sulfate	√	
Extraction/ExchangeSolvents: Methylene chloride, hexane, acetonitrile, acetone, pesticide quality or equivalent	√	√
Acetone, Methylene Chloride: Used for cleaning	√	√

7.2. Standards

7.2.1. Stock Standards

Stock standards are purchased as certified solutions. Semivolatile stock standards are stored according to manufacturer's instructions. All stock standards must be protected from light. Stock standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampoule is opened if purchased). Standards that are cold stored must be allowed to come to room temperature before use.

7.2.2. Surrogate Spiking Standards

Prepare or purchase surrogate spiking standards at the concentrations listed in Table 2. Surrogate spiking standards are purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light or stored according to manufacturer's instructions. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.3. Matrix Spiking and Laboratory Control Spiking Standards

The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Table 3. Spiking standards are purchased or prepared as dilutions of the stock standards.

Spiking solutions must be refrigerated and protected from light or stored according to manufacturer's instructions. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.4 See SOP NC-QA-017 for additional information on Standards and Reagents.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1 The holding time for solid and waste samples is 14 days from sampling to extraction.

8.3.2 Analysis of the extracts is completed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

9.1.1. The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS, and a matrix spike / matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS / MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD. See Policy QA-003 for further definition of the batch.

9.2. Sample Count

9.2.1. Laboratory-generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count. Field samples are included.

9.3. Method Blank (MB)

9.3.1. An MB consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the MB at the same level as the samples. See Table 2 for the appropriate amount of surrogate to use for each analytical method. The MB is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

9.3.2. Solid MB use approximately 30 g of sodium sulfate spiked with the surrogates. See Table 2 for the appropriate amount of surrogate to use for each analytical method. The MB goes through the entire analytical procedure.

9.4. Laboratory Control Sample (LCS)

9.4.1. LCSs are well-characterized laboratory-generated samples used to monitor

the laboratory day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure.

9.4.2. The LCS is made up in the same way as the MB (see Sections 9.4.1 through 9.5.2), but spiked with the LCS standard and the surrogates. See Table 3 for the appropriate amount of spike to use for each analytical method.

9.5. Surrogates

9.5.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.5.2. Each applicable sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. See Table 2 for the appropriate amount of surrogate spike to use for each analytical method.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1. An MS is an environmental sample to which known concentrations of target analytes have been added. An MSD is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and MS. See Table 3 for the appropriate amount of spike to use for each analytical method.

9.7. Initial Demonstration of Capability

9.7.1. The initial demonstration and method detection limit studies described in Section 13 must be acceptable before analysis of samples may begin.

9.8 Control Limits

9.8.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.8.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMs.

9.9 Method Detection Limits (MDLs) and MDL Checks

9.9.1 MDLs and MDL Checks are established by the laboratory as described in SOPs CA-Q-S-006 and NC-QA-021.

9.9.2 MDLs are easily accessible via the LIMs.

9.10 Nonconformance and Corrective Action

9.10.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action.

10. CALIBRATION AND STANDARDIZATION

10.1. On a weekly basis, measure the appropriate volume of solvent into the appropriate size glass vial using a gastight syringe that is manufactured to a certified volume delivery tolerance of ± 0.01 mL. The "standard" glass vial is sealed, and the meniscus is noted by marking a line on the bottle. The glass vials containing the sample extracts are then compared against the "standard" glass vial to ensure the final volume is consistently 1.0 ± 0.01 mL. A log is kept of the glass vial lot number and preparation date.

11. PROCEDURE

Refer to SOP NC-QA-016 for information on DoD samples.

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance memo and approved by a supervisor. The Nonconformance memo will be filed in the project file. Procedural variations are not allowed for Ohio VAP projects.

11.2 Soxhlet

11.2.1 Remove surrogate and matrix spiking solutions from refrigerator if cold stored, and allow to warm to room temperature.

11.2.2 Decant any water layer on a sediment/soil sample into the sample lid or Mason™ jar and return it into the sample after removing the amount needed for the extraction unless there are specific instructions not to decant. Record and document if a water layer was present in LIMS. Homogenize the sample by mixing it thoroughly in the container. If this is not possible, place the sample in a clean beaker and homogenize. Upon completion of homogenization in the beaker, return the sample to original container. Discard foreign objects such as sticks, leaves, and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials, consult with the client (via the Project Manager). If the

sample cannot be prepared using a Soxhlet due to matrix issues, a waste dilution may be required. Refer to SOP NC-OP-043 for the waste dilution procedure.

11.2.3 Place approximately 200mL of solvent into a 250 mL flat bottom flask containing one or two clean boiling chips. Weigh 30g ± 0.5 g of sample into a thimble or in a jar, recording the weight to the nearest 0.01g in LIMS. Sample weights less than 30g, but over 1g, may be used if the appropriate reporting limits can be met.

11.2.4 Prepare an MB, LCS, and MS/MSD for each batch as specified in Section 9 of this SOP, using sodium sulfate as the matrix for the LCS. The parent sample is used for the MS/MSD. The weight of sodium sulfate used must be approximately the weight of soil used in each sample.

11.2.5 Add anhydrous sodium sulfate to each sample and mix well. The mixture must have a free-flowing texture. If not, add more sodium sulfate. Add the sample/sodium sulfate mixture to a soxhlet extractor thimble, but do not pack the thimble tightly. The soxhlet extractor or extraction thimble must drain freely for the duration of the extraction period. A glass wool plug below the sample in the soxhlet extractor is an acceptable alternative for the thimble.

11.2.6 Add the appropriate amount of surrogate and matrix spiking solution as indicated in Tables 2 and 3.

11.2.7 Attach the flask to the extractor and extract the sample for 16-24 hours at 4-6 cycles per hour. Check the system for leaks at the ground glass joints after it has warmed up.

Note: If a reduced quantity of sample is extracted, it is usually necessary to increase the amount of sodium sulfate added or increase the solvent boiling rate to properly set the cycling rate.

Solvents:

Semivolatile GC/MS, TPH Organochlorine pesticides and PCBs	1:1 v/v Methylene Chloride / Acetone
8270 (MS) Concrete	Methylene Chloride
8082 Caulk Matrix	1:1 v/v Hexane / Acetone

11.2.8 Allow the extract to cool after the extraction is complete, then disassemble by gently twisting the soxhlet from the flask.

11.2.9 The sample is now ready for the concentration step (Section 11.5).

11.2.10 Cover with aluminum foil and refrigerate if the extract is not concentrated immediately. Refer to Section 11.5 for concentration.

11.3 Concentration: According to the type of sample, different solvents and final volumes will be required. Refer to Table 1 for the appropriate final volumes and concentrations.

11.3.1 Kuderna-Danish (KD) Method:

11.3.1.1 Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube (CT) to the 500 mL KD flask. Label the CT and KD. Transfer the sample to the labeled K-D flask, filtering Continuous Liquid/Liquid and Soxhlet samples through funnels filled with sodium sulfate. Rinse the funnel with 20-30 mL of methylene chloride to complete the quantitative transfer.

11.3.1.2 Add one or two clean boiling chips and the extract to be concentrated to the KD flask and attach a three-ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column. (This is important to ensure that the balls are not stuck, and the column will work properly). Attach to the KD flask.

11.3.1.3 Place the KD apparatus on a water bath (90-98°C) so the tip of the concentrator tube is submerged. The water level must not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter; but the chambers should not flood.

11.3.1.4 Concentrate to 15-20 mL. If the determinative method requires a solvent exchange, add the appropriate exchange solvent to the top of the Snyder Column, and then continue the water bath concentration back down to 5-8 mL. Refer to Table 1 for details of exchange solvents and final volumes. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.3.1.5 Remove the KD apparatus from the water bath and allow to cool for a minimum of 10 minutes. If the level of the extract is above the level of the CT joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

11.4 Nitrogen Evaporation to Final Concentration

11.4.1 Transfer the CT to the evaporation apparatus.

11.4.2 Place the tube in a warm water bath that is at least 5°C below the boiling

temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but must not create splattering of the extract.

Boiling points of commonly used solvents are:

Methylene chloride	40°C
Acetone	56°C
Hexane	69°C
Acetonitrile	82°C
Toluene	111°C

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.4.3 Refer to Table 1 to determine the final volume needed for a specific test method. Evaporate to slightly less than the required final volume.

Quantitatively transfer the extract to the appropriate final container and dilute to the appropriate final volume using the "standard" glass vial noted in Section 10.1. Cap the sample and affix the appropriate label. The sample is now ready for analysis.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

11.5 Analytical Documentation

11.5.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.5.2. Record all standards and reagents in the LIMS Reagents module. All standards and reagents are assigned a unique number for identification.

11.5.3. Record sample and associated QC information into LIMs. Level I and Level II technical reviews are performed in LIMS.

12. DATA ANALYSIS AND CALCULATIONS

12.1 Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

13.1.1. Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample

used to monitor method performance, which must contain all the analytes of interest. The spiking level must be equivalent to a mid-level calibration. (For certain tests, more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

13.1.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.1.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs. See SOPs NC-GC-038, NC-MS-018, NC-MS-003, and NC-GC-007 for detailed information on the determinative methods.

13.2. Training Qualification

13.2.1. The Group/Team Leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files

14. POLLUTION PREVENTION

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State, and local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15.2. The following waste streams are produced when this method is carried out.

15.2.1. Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride/acetone or acetone/hexane from the extract drying step. These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.2. **Assorted flammable solvent waste from various rinses**. These wastes are put into the halogenated/non-halogenated25 gallon solvent waste container located under the fume hood in extractions.

15.2.3. **Methylene chloride waste from various rinses:** These wastes are disposed of in the liquid-liquid separation unit.

15.2.4. **Hexane-Hexane waste:** These samples are to be disposed in the flammable waste.

15.2.5. **Waste Hexane in vials.** These vials are placed in the vial waste located in the GC prep laboratory.

15.2.6. **Waste Methylene Chloride sample vials**. These vials are placed in the vial waste located in the GC prep laboratory.

15.2.7. **Extracted solid samples contaminated with methylene chloride/acetone or acetone/hexane.** These materials are disposed of in the solid waste and debris in a red container located in the Extractions Lab.

15.2.8. Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCBs must be segregated into their own waste stream. PCB wastes are collected in one of three waste streams, solid PCB, liquid PCB, and PCB vial waste.

15.2.9. Solvent Recovery System Waste. Methylene Chloride waste from the Solvent Recovery System is collected and disposed of in the liquid-liquid separation unit. Acetone/Methylene Chloride waste from this system is disposed of in the flammable waste containers located in the laboratory.

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III (December 1996). Sections 3500B, 3540C, and 3580A

16.1.2. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.4 Corporate Quality Management Plan (CQMP), current version 16.1.5 Federal Register - Environmental Protection Agency, 40 CFR, Part 136, Volume 49, No. 209, October 26, 1984, Method 625

16.1.5. EPA 600, Methods for Chemical Analysis of Water and Wastes, Method 608

16.1.6. Revision History

Historical File:		Revision 3.4: 10/16/98		Revision 0: 03/12/08 (NC-OP-032)
(formerly CORP-OP-0001NC)		Revision 3.5: 04/22/99		Revision 1: 01/07/09 (NC-OP-032)
		Revision 3.6: 05/13/99		Revision 0: 03/24/11 (NC-OP-040)
		Revision 3.7: 03/20/01		Revision 1-A: 01/24/12
		Revision 3.8: 05/23/01		
		Revision 3.9: 04/22/02		
		Revision 4.0: 02/04/03		
		Revision 4.1: 10/07/03		
		Revision 4.2: 01/30/06		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5. Supplemental Practices for DoD Project Work SOP, NC-QA-016

16.2.6. Gas Chromatographic Analysis based on Method 8000B, 8021B, 8081A, 8081B, 8082, 8082A, 8151A, 8015B, 8015C, and 615, NC-GC-038

16.2.7. GC/MS Analysis based on Method 8270C and 8270D, NC-MS-018

16.2.8. Analysis of Pesticides and PCBs by EPA Method 608, NC-GC-007

16.2.9. GC/MS Semivolatile Organic Compounds Capillary Column Technique Based on EPA Method 625, NC-MS-003

16.2.10. Standards and Reagents, NC-QA-017

17. MISCELLANEOUS

17.1. Modifications from Reference method

17.1.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.1.2. Spiking levels for method 608 have been reduced by a factor of ten to bring the levels within the normal calibration range of the instrument.

17.2. Tables

TABLE 1

Final Volumes and Exchange Solvents

Type	Exchange Solvent for Analysis	Final Volume for Analysis in mL
Semivolatiles	N/A	2.0 mL
PCB	Approximately 18 mL Hexane – water Approximately 36 mL Hexane - solid	10.0 for solids
Pesticides	Approximately 18 mL Hexane	10.0 for solids
BNA – SIM	N/A	2.0 mL - Solids & H2O
TPH	N/A	5.0

Note: *Different final volumes may be necessary to meet special client reporting limit requirements.*

TABLE 2

Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume (mL)
BNA	20 ppm BNA	1.0
BNA / SIM	20 ppm BNA	0.1
PEST	0.2 ppm DCB/TCX	1.0
TPH	40ng Nonane (C9)	1.0
PCB	0.2 ppm DCB/TCX	1.0

TABLE 3
Matrix Spike and LCS Solutions

Analyte Group	Matrix Spike Solution ID	Volume (mL)
BNA	20 ppm BNA All-Analyte Spike and Restek Spike	1.0
BNA / SIM	20 ppm BNA All-Analyte Spike and Restek Spike	0.1
PEST	Pest NPDES Spike	1.0
PCB	10 ppm PCB Spike	1.0
TPH	See Spike List – Table 5	1.0

TestAmerica Canton

SOP Amendment Form

SOP NUMBER: **NC-WC-032, Rev. 9 Effective date: 3/21/13**

SOP TITLE: Cyanide Preparation Method

REASON FOR ADDITION OR CHANGE: Addition noted below

CHANGE EFFECTIVE FROM: (DATE): 11/21/13

Change(s) Made: Added the following to the SOP:

7.1.10 Sulfuric Acid: (H_2SO_4), 1:1

11.2.4.4 In the order stated, add 2.0 mL sulfamic acid solution, 5 mL of 1:1 sulfuric acid, and 2.0 mL of magnesium chloride solution to the distillation tube. Be sure to rinse the inlet tube sparingly with reagent water between and after reagent additions

EDITED BY/DATE: Lucas Grossman 11/21/13

*APPROVED BY:

Lucas Grossman
Technical Reviewer Signature Date: 11/21/13

Rebecca Strait
QA Manager Signature Date: 12/11/13

Technical Director Signature Date: 11/22/13

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Canton

SOP No. NC-WC-032, Rev. 9
Effective Date: 03/21/13

Title: CYANIDE PREPARATION METHOD

[Method: EPA Methods 335.1, 335.2, 335.2(CLP-M), 335.4, Standard Method 4500-CN-I, 4500-CN-E, 4500-CN-G, and SW846 Method 9012A, 9012B]

Approvals (Signature/Date):

Technology Specialist	3/21/2013 Date	Health & Safety Coordinator	3/21/2013 Date
Quality Assurance Manager	3/21/2013 Date	Laboratory Director	3/21/2013 Date

This SOP was previously identified as SOP No. NC-WC-032, Rev 8.8-A, dated 04/16/12

Facility Distribution No. _____ **Distributed To: _____**

TABLE OF CONTENTS

1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Total, Amenable, and Weak Acid Dissociable in solids, liquids, and waters. It is based on SW 846 Method 9012A, 9012B, EPA 335.1, 335.2, 335.2 (CLP-M) Ohio VAP only, 335.4, and Standard Methods 4500-CN-E,4500-CN-G and 4500-CN-I. The working linear range is 0.005 - 0.2 mg/L for waters and 0.25 to 10 mg/kg for solids.

1.2. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory.

2. SUMMARY OF METHOD

2.1. The Cyanide, as HCN, is released by distilling/refluxing the sample with strong acid and is trapped in a sodium hydroxide solution.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current version. Refer to Appendix II at the end of this document for a glossary of acronyms.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. All glassware is cleaned per SOP NC-QA-014. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Interferences are eliminated or reduced by using the distillation procedure. Oxidizing agents, such as chlorine, will decompose most cyanides. Sulfide will distill over with the cyanide and could affect colorimetric, titrimetric, and electrode procedures. Refer to the preparation section on how to screen and treat samples appropriately. The possibility of interferences from nitrate and nitrite is eliminated by pretreatment with sulfamic acid just before distillation.

4.3. Aldehydes convert cyanide to cyanohydrin which could result in the loss of cyanide. If the presence of aldehydes is suspected, stabilize the sample with NaOH at the time of collection and add 2 mL 3.5% ethylenediamine solution per 100 mL of sample.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental

Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. In the event the sample begins to react unexpectedly during distillation, remove entire apparatus from heat source, set aside, and allow to cool. **DO NOT ATTEMPT TO DISASSEMBLE GLASSWARE.** Doing so may result in a sudden release of pressure with spraying of the sample.

5.3. Latex, vinyl, nitrile, or similar gloves may be used.

5.4. Preparation of sodium hydroxide solutions produces considerable amounts of heat. Use plastic containers to mix this solution if possible. If glass containers are used, they must be free of any cracks or irregularities.

5.5. The acidification of samples prior to extraction/preparation can result in the release of a highly toxic gas, hydrogen cyanide.

5.6. If samples are identified with cyanide concentrations equal to or greater than 200 mg/L, immediately notify the Department Manager and personnel responsible for hazardous waste shipping. Those samples must be identified as extremely hazardous for other chemists and must receive special attention during disposal. **Potassium cyanide and sodium cyanide will give off Hydrogen Cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.**

5.7. Cyanide and cyanide salts are extremely toxic. Addition of acid can generate hydrogen cyanide gas, which can be extremely dangerous.

5.8. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Potassium Cyanide	Poison Corrosive	5 mg/m^3 TWA as CN	**This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heart beat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred

			vision, and eye damage.
Ascorbic Acid	Slight Health, Reactivity, Flammability, and Contact	No PEL Est	May cause mild irritation to the respiratory tract
Acetic Acid (1)	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Cadmium Carbonate	Probable carcinogen	0.01 mg/ m^3 as Cd	Ingestion causes increased salivation, choking, vomiting, stomach pains and diarrhea. Inhalation may cause respiratory irritation, nausea and dyspnea.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3 2 mg/m^3-Ceiling	This material will cause burns if comes into contact with the skin or eyes. Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Magnesium Chloride	Health	No PEL est.	Inhalation of dust may cause mild irritation to the mucous membranes.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Sulfamic Acid	Corrosive Poison	No PEL est.	Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. Can be fatal if ingested.
Sodium Cyanide	Poison Corrosive	5 mg/m^3 TWA as CN (skin)	**This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosiveto the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heartbeat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed

			through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Zinc Acetate	Irritant	None Listed	Symptoms of skin or eye contact include redness, itching and pain.
Silver Nitrate	Corrosive Poison Oxidizer	0.01 mg/m^3 (TWA) for silver metal dust and fume as 0.02 Ag	This is a corrosive, poisonous material. It will cause burns to any area of contact and is harmful if inhaled. Ingestion may cause death. Contact with other material may cause fire. Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. May be absorbed into the body following inhalation. Swallowing can cause severe burns of the mouth, throat and stomach. Can cause sore throat, vomiting, and diarrhea. Poison. Symptoms include pain and burning in the mouth, blackening of the skin and mucous membranes, throat, and abdomen, salivation, vomiting of black material, diarrhea, collapse, shock, coma and death. Skin contact can cause redness, pain and severe burns. Eye contact can cause blurred vision, redness, pain, severe tissue burns and eye damage.
1 – Always add acid to water to prevent violent reactions.			

5.9 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.10 Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.11 The preparation of standards and reagents must be conducted in a fume hood with the sash closed as far as the operation will permit.

5.12 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Cyanide Distillation Apparatus

6.2. Analytical balance, capable of accurately weighing ? 0.0001 g

6.3. Vacuum source

6.4. Calibrated Class B Disposable Beakers: 50 mL

6.5. Volumetric flasks: 100 mL, 200 mL,500 mL, 1 L

6.6. Volumetric pipettes: range from 0.01 to 20 mL

6.7. Balance: Top loading, capable of accurately weighing ? 0.01 g

6.8. Lead Acetate Indicator Paper

6.9. Potassium Iodide (KI) Indicator Paper

6.10. Buret: Class A 10 mL

6.11. pH strips

6.12. Snap seal containers: 120 mL

6.13 Plastic bottles with lids: 250mL or 500mL

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Sulfamic Acid: reagent grade

7.1.2. Sulfamic Acid Solution: Add 100 g of sulfamic acid to 800 mL reagent water and dilute to 1 liter with reagent water

7.1.3. Ascorbic Acid: reagent grade

7.1.4 Sodium Thiosulfate: reagent grade

7.1.5 Sodium Thiosulfate Solution (0.02 g/L)

7.1.6 Sodium Thiosulfate Solution (500 g/L)

7.1.7 Hydrogen Peroxide (H2O2) (3%): purchased.

7.1.8 Sodium Hydroxide: (NaOH), high purity grade.

7.1.9 Sodium Hydroxide, 0.25 N: Add 10 g of NaOH to 900 mL reagent water and dilute to 1 liter with reagent water. Purchased reagent may also be used.

7.1.10 Sulfuric Acid: (H_2SO_4), 1:1

7.1.11. Acetic Acid Solution: Add 200 mL of reagent water to a 1.0 L volumetric flask.

Carefully add 750 mL of glacial acetic acid, and bring to volume with reagent water. Transfer to a 1.0 L amber bottle and cap.

7.1.12. Magnesium Chloride: ($MgCl_2?6H_2O$), reagent grade.

7.1.13. Magnesium Chloride Solution: Add 510 g $MgCl_2?6H_2O$ to 500 mL reagent water and dilute to 1 liter with reagent water. Purchased reagent may also be used.

7.1.14. Calcium Hypochlorite: ? Ca $(OC1)_2$? , reagent grade

7.1.15. Calcium Hypochlorite Solution: Add 5 g of $Ca(OC1)_2$ to 100 mL of reagent water. Store in an amber glass bottle in the dark. Prepare monthly.

7.1.16. Methyl Red Indicator: Add 0.25 g of methyl red to 250 mL of glacial acetic acid and dilute to 500 mL with reagent water.

7.1.17. Methyl red reagent grade glacial

7.1.18. Acetic Acid: (CH_3COOH), glacial reagent grade

7.1.19. Zinc Acetate: ? $Zn(C_2H_3O_2)$? , reagent grade

7.1.20. Zinc Acetate Solution: Add 100 g zinc acetate to 800 mL reagent water and dilute to 1 liter with reagent water.

7.1.21. Sodium Acetate: ? $NaC_2H_3O_2?3H_2O$? reagent grade

7.1.22 Sodium Acetate Buffer: Add 410 g of sodium acetate to 500 mL of reagent water. Adjust the pH to 4.5 using glacial acetic acid and dilute to 1 liter with reagent water.

7.1.23. Rhodanine: reagent grade

7.1.24. 0.0192 N Silver Nitrate: reagent grade

7.1.25. Cadmium carbonate [$CdCO_3$]: reagent grade

7.2. Standards

7.2.1. Primary Source Cyanide Stock Standard, 1000 mg/L: Add 2.51 g of potassium cyanide (KCN) and 2.0 g of potassium hydroxide (KOH) to a 1000 mL volumetric flask and dilute to volume with reagent water. Mix well and store in glass amber container. Stable for 1-3 months. Additional information can be found in SOP NC-QA-017.

Note: This stock standard may also be purchased.

Note: Prepared stock standard must be standardized prior to use. See

7.2.2. Secondary Source Cyanide Standard, 1000 mg/L: Follow Section 7.2.1 using an alternate source of Potassium Cyanide (KCN).

Note: This stock standard may also be purchased.

Note: Prepared stock standard must be standardized prior to use. See Appendixl.

7.2.3. Calibration Standards (Water and Solid Matrices)

7.2.3.1 If using the purchased 0.25N NaOH: Pipette the appropriate amount of cyanide standard into 100 mL volumetric, and bring to volume with 0.25N NaOH. Prepare weekly.

Concentraction CN-	ML CN-	Final Volume
10 mg/L	10 mL of 100 mg/L	100 mL
1.0 mg/L	20 mL of 10 mg/L	200 mL

8. SAMPLE PRESERVATIONAND STORAGE

8.1. Solid and liquid samples are not chemically preserved. Water samples are preserved with NaOH to a pH?1 2. All samples are stored at 4? C ? 2? C in plastic or glass containers.

8.2. The holding time for samples is 14 days from sampling to analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (Laboratory Control Sample Method Blank, Matrix Spike , Matrix Spike Duplicate) which are processed similarly with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same process.

9.2. Method Blank (MB)

9.2.1. One method blank must be processed with each preparation batch. The method blank must contain all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the

analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank must not contain any analyte of interest at or above the reporting limit.

9.2.1.1. A method blank (MB) consists of 50 mL 0.25N NaOH for Total Cyanide analysis or 50 mL reagent water for Weak Acid Dissociable and Amenable Cyanide analysis. The method blank (MB) for solids consists of 50 mL 0.25N NaOH or reagent H_2O and 1.0g Ottawa sand. The method blank (MB) must be distilled per Section 11.4.3 and analyzed with each analytical batch of samples.

9.2.2 Corrective Action for Method Blanks

9.2.2.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are re-prepared and re-analyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.2.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One Laboratory Control Sample must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The laboratory control sample is used to monitor the accuracy of the analytical process. Ongoing monitoring of the laboratory control sample results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. For Method 335.4, laboratory control sample recoveries must be within 90% to 110%, or corrective action is required.

9.3.2. A mid-range laboratory control sample consisting of a 0.04 mg/L (2.0 mL of 1.0 mg/L secondary source to 50 mL) must be distilled per Section 11.4.3 and analyzed with each analytical batch of samples for Total, Non-Amenable, and Weak Acid Dissociable Cyanide analysis.

Note: A purchased complex cyanide solution may be used instead as the mid-range laboratory control sample for **Total Cyanide** analysis only.

9.3.3. Corrective Action for Laboratory Control Samples

9.3.3.1. If any analyte is outside established control limits, the system is out of control and corrective action must occur.

9.3.3.2. Corrective action must be re-preparation and re-analysis of the batch unless the client agrees that other corrective action is acceptable. For

Ohio VAP samples, the laboratory control sample must be re-distilled, unless the laboratory control sample is biased high and the samples are non-detect.

9.3.3.3. The only exception is that if the laboratory control sample recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative**.

9.4 Additional information on QC samples can be found in QA Policy QA-003.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.5.1 One matrix spike/matrix spike duplicate pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to matrix spike/matrix spike duplicates. The matrix spike duplicate results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for matrix spike/matrix spike duplicate analysis.

9.5.1.1 A matrix spike/matrix spike duplicate consisting of 50 mL or 1.0g of sample brought up to 50 mL with reagent water and 2.0 mL of the 1.0 mg/L of secondary standard must be distilled with each analytical batch of samples. For samples requiring Amenable analysis, a matrix spike/matrix spike duplicate consisting of 50 mL or 1 g of sample brought up to 50 mL with reagent water must be prepared and carried through all steps of the Amenable process. After distillation, the distillate is spiked with 2 mL of the 1ppm standard. The same sample must have a total cyanide matrix spike/matrix spike duplicate performed. The difference is calculated and reported.

9.5.2 Corrective action for Matrix Spike/Matrix Spike Duplicates

9.5.2.1 If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the laboratory control sample. If the laboratory control sample recovery is within control limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the laboratory control sample is outside control limits, corrective action must be taken. Corrective action must include re-preparation and re-analysis of the batch.

9.5.2.2 If the native analyte concentration in the matrix spike/matrix spike duplicate exceeds four times the spike level for that analyte, the recovery

data is flagged with a "4" in LIMS.

9.6 Control Limits

9.6.1 Control limits are established by the laboratory as described in SOP NC-QA-018, with the exception of Method 335.4 which has stated control limits for the laboratory control sample, matrix spike, and matrix spike duplicate of 90% to 110%.

9.6.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMs

9.7 Method Detection Limits (MDLs) and MDL Checks

9.7.1 MDLs and MDL Checks are established by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.7.2 MDLs are easily accessible via LIMs.

9.8 Non-conformance and Corrective Action

9.8.1 Any deviations from QC procedures must be documented as a non-conformance, with applicable cause and corrective action.

10. CALIBRATION AND STANDARDIZATION

10.1 A low and high standard are distilled from the same source as the calibration curve each day. Prepare the low standard (0.025 mg/L) by diluting 0.125 mL of 10 ppm standard with 0.25N NaOH to a final volume of 50 mL. Prepare the high standard (0.1 mg/L) by diluting 0.5 mL of 10 ppm standard with 0.25N NaOH to a final volume of 50 mL. If distilled standards do not agree within 10% of the undistilled standards, the analyst must find the cause of the apparent error before proceeding. The instrument is calibrated daily using the 0.2 ppm standard (see SOP NC-WC-031), which is diluted by the instrument into the following concentrations: 0.005, 0.010, 0.025, 0.050, 0.100, 0.200.

11. PROCEDURE

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Non-conformance Memo. The Non-conformance Memo must be filed in the project file. This is not applicable for Ohio EPA/VAP.

11.2 Sample Preparation Procedure applicable for use with the Midi distillation unit.

11.2.1 For DoD work, refer to SOP NC-QA-016 for specific details.

11.2.2 Checking for Interferences

11.2.2.1 Using pH paper strips, check the pH of the sample and document in the LIMS worksheet if the ph is <12. If pH is <12, the deviation **must be addressed in the project narrative.**

11.2.2.2 Test each sample for the presence of sulfides using lead acetate paper. If sulfides are present, treat the sample with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution. Avoid a large excess of cadmium carbonate and long contact time in order to minimize loss by complexation or occlusion of cyanide on the precipitated material. **If any sample in a batch is treated, the Method Blank (MB) and Laboratory Control Sample (LCS) for the batch must undergo the same treatment.** Document this in LIMS. NOTE: If Sulfide is present, and more than 48 hours have elapsed since sampling, the analyst must create an NCM notifying the client of the presence of Sulfide.

11.2.2.3 Test each sample for the presence of chlorine using potassium iodide test strips. If chlorine is present, add small portions of sodium thiosulfate solution (0.02 g/L) with constant re-testing until the oxidizers are neutralized. Avoid any excess thiosulfate solution. For method 335.4 add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper.

11.2.3 Amenable Cyanide (Chlorinated Aliquot)

11.2.3.1 Place 50 mL (waters) or 1.0 g (solids/liquids) into a snap cap container. Add 50 mL of reagent water to non-waters. Place the snap cap container on a stir plate and begin mixing. Test the pH of the solution. If less than 12, add 1.25 N NaOH, drop by drop until pH ? 12. Drop by drop, add calcium hypochlorite until an excess of chlorine is reached. Test for chlorine excess using KI paper. Allow the sample to chlorinate for one hour maintaining excess chlorine and pH between 11 and 12. If necessary, add more Ca(OCL)2 and/or NaOH. Either cover snap cap containers with aluminum foil, or place box over snap cap containers to keep them in the dark during the chlorination process. At the end of the chlorination period, add 8 drops of H2O2 (3%) followed by 4 drops Na2S2O3 solution (500g/L) to destroy excess chlorine. Test using KI paper until there is no color change. Pour the sample into a distillation flask and follow the total cyanide preparation method (Section 11.4.3). Also set up an unchlorinated aliquot of sample (50 mL or 1.0 g) following the total cyanide method. **Reminder**: Do not use the purchased complex cyanide standard for the Amenable LCS.

11.2.4 Total Cyanide

11.2.4.1 Add 50 mL or 1.0 g of the sample to the distillation tube. Bring the final volume to 50 mL with reagent water. Add 25 mL of 0.25 N NaOH

solution to the collection trap. Assemble the cyanide distillation apparatus.

11.2.4.2 Waste Procedure - Sometimes waste samples will stick to the weigh pan making it difficult to rinse all of the sample into the distillation tube, causing a biased low result. In this case, aliquot the sample onto a Whatman 47mm filter instead of a weigh pan. If the sample is bleeding through the filter, use extra filters to prevent sample loss due to over-saturation of the filter. It may be helpful to form the filter into a bowl shape before aliquoting to keep the sample from running off the filter. Once the sample is weighed, the filter can be wrapped around the sample and dropped into the distillation tube. When preparing waste samples in this way, an extra blank must be distilled with a filter added to the distillation tube. This blank must be analyzed and shown to be free of cyanide to prove the filters do not contain cyanide. Proceed with the rest of the distillation as usual.

11.2.4.3 Turn on the vacuum source and adjust the flow such that the bubbles in the collection trap are steady (~10 bubbles/second) but not bubbling so fast that the NaOH is splashing in the collection trap. At this time, add any spiking solutions to the LCS or MS/MSD samples directly into the inlet tube.

11.2.4.4 In the order stated, add 2.0 mL sulfamic acid solution, 5 mL of 1:1 sulfuric acid, and 2.0 mL of magnesium chloride solution to the distillation tube. Be sure to rinse the inlet tube sparingly with reagent water between and after reagent additions.

11.2.4.5 Flip the heater switch to "on" to allow the apparatus to warm up. Be sure to adjust the air flow and water as necessary. Heat for one hour. Allow to cool for 15 minutes, while keeping the vacuum on.

11.2.4.6 Disconnect the absorber. Pour solution into a 120 mL snap seal container or place the collection trap into designated Styrofoam holders. Check the pH of the solution to ensure it is >12. **NOTE:** If the pH is not >12, do NOT remove the sample from the hood. Contact the Technical Director, Operations Manager, or EH&S representative immediately for further instruction. Do not rinse the scrubber tube or dilute NaOH in the snap seal. Be sure to properly label the bottles "total" or "amenable" along with sample ID, position, and date.

11.2.5 Weak Acid Dissociable

11.2.5.1 Add 50 mL or 1.0 g of sample to the distillation tube. Bring all volumes up to 50 mL with reagent water. Add 25ml 0.25N NaOH solution to the collection trap and assemble.

11.2.5.2 Turn on the vacuum source. Add any spiking standards to the appropriate LCS or MS/MSDs at this time. Through the inlet tube, add

1.0 mL of sodium acetate buffer, 1.0 mL of zinc acetate, and 0.25 mL of methyl red indicator. Rinse the inlet tube sparingly with reagent water between and after reagent additions. If the sample is not red, carefully add 75% acetic acid drop-wise until the sample does turn red. Check the sample color periodically throughout the distillation hour to ensure the sample stays red. **Reminder:** Do not use the complex purchased cyanide solution standard as the LCS for Weak Acid Dissociable cyanide analysis.

11.2.5.3 Flip on the heater switch (this allows the apparatus to warm up). Adjust the air flow and water as needed. Heat for one hour. Allow the sample to cool for 15 minutes with the vacuum on...

11.2.5.4 Pour the scrubber contents into a 120 mL snap seal bottle or place collection traps in designated Styrofoam holders. Do not rinse the scrubber. Label the bottle well, and be sure to denote it is a Weak Acid Dissociable sample distillate. Dispose of addition tubes used during WAD CN distillation to avoid contamination due to complex CN.

11.2.6 Cyanide Unit Cleanup

11.2.6.1 Cyanide distillation unit glassware is very fragile and expensive. It must be handled with care at all times.

11.2.6.2 Disassemble each setup. Be sure to collect the solids in a screen and dispose of properly.

11.2.6.3 Wash each setup with hot water. Rinse several times with reagent water.

Note: After preparing samples found to be extremely high in cyanide, distill 0.25 N NaOH through the system.

11.2.6.4 Re-assemble the setup.

11.2.6.5 Be sure to wash each setup as a separate unit, and replace in the same position. Wait for cyanide results before cleaning the glassware.

11.2.6.6 If a sample of known high cyanide concentration was distilled in a certain position, be sure to change the appropriate tubing on that position. Dispose plastic inlet tubes on any sample that was a hit for cyanide and on all weak acid dissociable samples. Some samples may be non-detect for weak acid, but have a high concentration of complex cyanide that is not released unless total reagents are used.

11.3 Analytical Documentation

11.3.1 Record all analytical information appropriately in LIMS, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or

modifications to the method.

11.3.2 All standards are logged into the LIMS reagent module. All standards are assigned a unique number for identification.

11.3.3 Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.3.4 Sample results and associated QC are transferred directly into LIMS from the instrument. Level I and Level II review is done in LIMS.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1 All waste must be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2 Waste Streams Produced by the Method

15.2.1 The following waste streams are produced when this method is carried out.

15.2.1.1 Total cyanide waste is disposed of in the designated container labeled "Acid Waste". Weak Acid dissociable waste is disposed of in a designated container labeled "Weak Acid Dissociable Waste".

15.2.1.2 Standard Waste and High Concentration Samples: This waste is disposed of in the designated container labeled "High Cyanide/Basic Waste." NO ACID is added to this container.

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1 References.

16.1.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Total and Amenable Cyanide, Automated UV; Method 9012A, Revision 1, 1996

16.1.2 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Total and Amenable Cyanide, Automated UV; Method 9012B, Revision 2, 2004

16.1.3 EPA 600; Cyanide, Total and Cyanide, Amenable to Chlorination; Methods 335.1, 335.4, 335.2, and 335.2(CLP-M)

16.1.4. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition; Weak Acid Dissociable Cyanide; Method 4500-CN-I

16.1.5. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition; Complex Cyanide; Method 4500-CN-E, 4500 CN-G

16.1.6. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition; Colorimetric Method; Method 4500-CN-E, 4500 CN-E

16.1.7. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.8 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.9 Corporate Quality Management Plan (CQMP), current version

16.1.10 Ohio EPA Laboratory Manual for Chemical Analyses of Public Drinking Water,

2000

16.1.11 Revision History

Historical File:		Revision 7.0: 03/19/98		Revision 8.4: 12/08/04
		Revision 8.0: 02/04/99		Revision 8.5: 05/30/08
		Revision 8.1: 03/20/00		Revision 8.6: 12/30/08
		Revision 8.2: 05/31/01		Revision 8.7: 06/16/10
		Revision 8.3: 06/17/03		Revision 8.8-A: 4/16/12

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

C:\Documents and Settings\fullerma.TA\Local Settings\Temporary Internet Files\Content.Outlook\AppData\Local\Microsoft Windows\POLICY16.2.2 Glassware Washing, NC-QA-014

16.2.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4 Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5 Supplemental Practices for DoD Projects, NC-QA-016

16.2.6 Standards and Reagents, NC-QA-017

16.2.7 Cyanide, Automated Method, NC-WC-031

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Method Deviation [9012A, 335.1, 335.2, 335.2(CLP-M) Ohio VAP only]

17.1.1. The reflux distillation apparatus used is the midi distillation.

17.1.2. The volume of sample used is reduced to 50 mL vs. 500 mL using the midi-distillation apparatus.

17.1.3. Samples are refluxed for 1 hr instead of 1.5 hrs per 335.2 and 335.4

17.1.4. Cadmium Carbonate and Sodium Thiosulfate are used to treat sample interferences except for method 335.4.

17.1.5. Method of Standard Addition is not performed for samples with matrix interference (sulfides).

17.1.6. Solid and waste samples are analyzed according to 335.2(CLP-M).

17.1.7. According to Method 335.2 (CLP-M) section 7.2.2.1 a mid-range standard must be distilled and compared to the curve for each SDG [batch] with a recovery of ±15%. The laboratory distills a high and low standard daily with a recovery requirement of ±10%.

Appendix I

CYANIDE STANDARDIZATION

1. Pipette 10.0 mL of the 1000 ppm stock cyanide standard into a 250 mL Erlenmeyer flask and add 90 mL of reagent water.

2. Add 0.5 mL (10 drops) of Rhodanine indicator.

3. Titrate with 0.0192 N silver nitrate (using a micro burette) until the color changes from yellow to pink/orange.

4. Titrate a blank (100 mL reagent water) following Steps 2 and 3.

5. Calculation

$$\text{Cyanide, mg / L} = \frac{\text{(A - B) (1000)}}{\text{mL Cyanide Solution (10)}}$$

Where:
A = mL titrant for standard
B = mL titrant for blank

6. If the cyanide concentration is not 1000 ppm, adjust concentration accordingly.

Appendix II

GLOSSARY OF ACRONYMS

CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CQMP	Corporate Quality Management Plan
DOC	Demonstration of Capability
DOD	Department Of Defense
DUP	Duplicate
EH&S	Environmental Health and Safety
ICB	Initial Calibration Blank
ICV	Initial Calibration Verification
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
MB	Method Blank
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheet
NCM	Non Conformance Memo
OSHA	Occupational Safety and Health Administration
OVAP	Ohio Voluntary Action Program
PEL	Permissible Exposure Limit
QAM	Quality Assurance Manual
QA/QC	Quality Assurance/Quality Control
RPD	Relative Percent Difference
SOP	Standard Operational Procedure
STEL	Short Term Exposure Limit
WAD	Weak Acid Dissociable

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

West Sacramento

SOP No. WS-IDP-0005, Rev. 1.5
Effective Date: 12/21/2012
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`Title: Preparation of Samples for Analysis of Polychlorinated Dioxins and Furans for Analysis HRGC/HRMS

[Methods 8290, 8290A & TO-9A]

Approvals (Signature/Date):

Elizabeth Nguyen Technical Manager 12/21/12 Date	Joe Schairer Health & Safety Manager / Coordinator 12/20/12 Date
Douglas Weir Quality Assurance Manager 12/20/12 Date	Karla Buechler Laboratory Director 12/21/12 Date

Facility Distribution No. _____ Distributed To: _____

1. SCOPE AND APPLICATION

1.1. This method provides procedures for the preparation of samples prior to the analysis of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD), polychlorinated dibenzo-p-dioxins (tetra- through octachlorinated homologs; PCDDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologs; PCDFs) in a variety of environmental matrices at part-per-trillion (ppt) concentrations by SW 846 Method 8290. The analytical method calls for the use of high-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. Refer to Table 1 for the list of analytes. Analysis is by SOP WS-ID-0005.

1.2. The sensitivity of this method is dependent upon the level of interferences within a given matrix.

1.3. This method is designed for use by analysts who are experienced with residue analysis.

1.4. Samples containing concentrations of specific congeners (PCDDs and PCDFs) that are greater than the calibration limit should be analyzed by a protocol designed for such concentrations, such as 8280A/B.

2. SUMMARY OF METHOD

2.1. This procedure uses matrix-specific extraction and analyte-specific cleanup techniques.

2.2. A specified amount (see Table 1) of soil, sediment, fly ash, water, sludge (including paper pulp), still-bottom, fuel oil, chemical reactor residue, air sample (QFF, PUF or XAD media) or fish tissue, is spiked with a solution containing specified amounts of each of nine isotopically (^{13}C) labeled PCDDs/PCDFs listed in Table 2. The sample is then extracted according to a matrix-specified extraction procedure. The extraction procedures are: a) toluene Soxhlet (or equivalent) extraction, for soil, sediment, fly ash samples, aqueous sludges, and solid air matrices (XAD, QFF, PUF); b) methylene chloride liquid-liquid extraction or solid phase extraction for water samples; c) dilution of a small sample aliquot in solvent for wastes/chemical products; and d) toluene (or hexane/methylene chloride) Soxhlet (or equivalent) extraction for fish tissue. This method can also use solid phase extraction (SPE), however, Test America West Sacramento is in the developmental stages for this extraction type and is not currently certified for its use.

2.3. If interferences are present, extracts may be cleaned as described below. The extracts are submitted to an acid and/or base washing treatment and dried. Following a solvent exchange step, the residue is cleaned up by column chromatography on acid/base silica, acid alumina and carbon on silica. The preparation of the final extract for HRGC/HRMS analysis is accomplished by adding 20 µL of a tetradecane solution containing 100 pg/µL of each of the two recovery standards $^{13}C_{12}$-1,2,3,4-TCDD and

$^{13}C_{12}$ -1,2,3,7,8,9-HxCDD (Table 2) to the concentrated eluate. The former is used to determine the percent recoveries of tetra- and penta-chlorinated PCDD/PCDF internal standards while the latter is used for the determination of hexa-, hepta- and octa-chlorinated PCDD/PCDF internal standard percent recoveries. Upon client approval, less final volume can be used to decrease detection limit and more final volume can be used to decrease severe interferences.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

3.3. Internal Standard: An internal standard is a ^{13}C-labeled analog of a congener chosen from the compounds listed in Table 2. Internal standards are added to all samples including method blanks and quality control samples before extraction, and they are used to quantitate the concentration of the analytes. Nine internal standards are used in this method. There is one for each of the dioxin and furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight. Additional internal standards may be added to act as retention time references, but they are not used for quantitation.

3.4. Recovery Standard: Two recovery standards are used to determine the percent recoveries for the internal standards. The $^{13}C_{12}$-1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated internal standards while $^{13}C_{12}$-1,2,3,7,8,9-HxCDD is used to determine the recovery of the hexa-hepta- and octachlorinated internal standards. $^{13}C_{12}$-1,2,3,7,8,9-HxCDD also acts as a retention time reference for the unlabeled analog present in sample extracts. They are added to the final sample extract before HRGC/HRMS instrument analysis.

3.5. Cleanup Recovery Standard (CRS): A $^{37}Cl_4$-2,3,7,8-TCDD analog that is added to each sample following extraction to measure the efficiency of the cleanup process.

4. INTERFERENCES

4.1. Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferents under the conditions of analysis by running laboratory method blanks. Analysts shall not use PVC gloves.

4.2. The use of high-purity reagents and solvents helps minimize interference problems. Purification of solvents by distillation in all-glass systems may be necessary.

4.3. Proper cleaning of glassware is extremely important because glassware may not only contaminate the samples, but may also remove the analytes of interest by adsorption on the glassware surface.

4.3.1. Glassware should be rinsed with solvent and washed with a detergent solution as soon after use as is practical. Sonication of glassware containing a detergent solution for approximately 30 seconds may aid in cleaning. Glassware with removable parts, particularly separatory funnels with Teflon stopcocks, must be disassembled prior to detergent washing.

4.3.2. After detergent washing, glassware should be immediately rinsed with acetone, toluene, hexane, and then methylene chloride.

4.3.3. Do not kiln reusable glassware in an oven as a routine part of cleaning. Kilning may be warranted after particularly dirty samples are encountered, but should be minimized, as repeated kilning of glassware may cause the formation of active sites on the glass surface that will irreversibly adsorb PCDDs/ PCDFs.

4.3.4. Immediately prior to use, Soxhlet (or equivalent) extraction glassware should be pre-extracted with toluene for a minimum of 3 hours. Note: Accelerated extractors such as the Soxtherm can use a shorter cleaning cycle which exhibits subsequent extractions free of cross contamination and interferences.

Note: Re-use of glassware should be minimized to avoid the risk of contamination. All glassware that is re-used must be scrupulously cleaned as soon as possible after use, applying the following procedure:

4.4. Interferences co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the PCDDs and PCDFs. The most frequently encountered interferences are chlorinated-biphenyls, methoxy biphenyls, hydroxy biphenyl ethers, benzyl phenyl ethers, polynuclear aromatics, and pesticides. Because very low levels of PCDDs and PCDFs are measured by this method, the elimination of interferences is essential. The cleanup steps given in Sections 11.12 thru 11.16 can be used to reduce or eliminate these interferences.

4.4.1. If South Carolina samples show diphenyl ethers at levels that could contribute to positive furan hits, a subsequent clean-up to remove them must be performed.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the West Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toes, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Hearing protection must be worn when using mechanical systems to grind fish, tissue, or paper/pulp samples.

5.1.2. Finely divided dry soils contaminated with PCDDs and PCDFs are particularly hazardous because of the potential for inhalation and ingestion. Such samples are to be processed in a confined environment, such as a hood or a glove box.

5.1.3. Assembly and disassembly of glassware creates a risk of breakage and cuts. All staff members shall wear Kevlar or MAPA blue latex cut-resistant gloves over chemically resistant gloves when assembling and disassembling glassware.

5.1.4. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile or similar gloves must be used. Latex gloves may be used for methanol.

5.1.5. Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.6. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.7. The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. The use of separatory funnels during the partition and back extraction of sample extracts can also create excessive pressure. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggles when it is performed. Alternately, the extraction can be performed behind a closed fume hood sash on a mechanical shaker.

5.1.8. When Dean-Stark/Soxhlet clean-ups or extractions are performed overnight or unattended, special precautions must be taken. Open the chiller valves to the system about 15 minutes before the heating elements are turned on, and check every condenser to ensure that it is cold and functioning properly before turning the heating elements on. Check every condenser again about 15 minutes after turning the heating elements on to ensure that they are still cold and functioning properly. If the system is left operating overnight or unattended for an extended period, the first chemist to come back into the lab must again check every condenser to ensure that it is still cold and functioning properly.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Benzene	Flammable Toxic Carcinogen	PEL: 1 ppm TWA ; 5 ppm 15 MIN. STEL	Causes skin irritation. Toxic if absorbed through skin. Causes severe eye irritation. Toxic if inhaled. Vapor or mist causes irritation to mucous membranes and upper respiratory tract. Exposure can cause narcotic effect. Inhalation at high concentrations may have an initial stimulatory effect on the central nervous system characterized by exhilaration, nervous excitation and/or giddiness, depression, drowsiness or fatigue. Victim may experience tightness in the chest, breathlessness, and loss of consciousness.
Cyclohexane	Flammable Irritant	300 ppm TWA	Inhalation of vapors causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. High concentrations have a narcotic effect.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Isooctane	Flammable Irritant	None established	Inhalation of vapors may cause nausea, headache, dizziness, loss of consciousness, irritation to upper respiratory tract, pain in throat and nose, coughing, wheezing, shortness of breath.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Tetradecane	Irritant	None established	Inhalation of vapors may cause difficulty breathing, headache, intoxication and central nervous system damage.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

The following list of items does not necessarily constitute an exhaustive compendium of the equipment needed for this analytical method.

6.1. Nitrogen evaporation apparatus with variable flow rate.

6.2. Balances capable of accurately weighing to 0.01 g and 0.0001 g.

6.3. Centrifuge.

6.4. Water bath, equipped with concentric ring covers and capable of maintaining temperature control within ± 2°C.

6.5. Stainless steel or glass containers large enough to hold contents of one-pint sample containers.

6.6. Drying oven.

6.7. Stainless steel spoons and spatulas.

6.8. Pipettes, disposable, Pasteur, 150 mm long x 5 mm ID.

6.9. Pipettes, disposable, serological, 10 mL, for the preparation of the carbon column specified in Section 7.1.

6.10. Reacti-vial, 2 mL, silanized clear glass.

6.11. Stainless steel meat grinder with a 3- to 5-mm hole size inner plate.

6.12. Separatory funnels, 250 mL.

6.13. Separatory funnels, 1000 mL.

6.14. Teflon® boiling chips (or equivalent) washed with methylene chloride before use.

6.15. Chromatographic column, glass, 300 mm x 10.5 mm, fitted with Teflon® stopcock.

6.16. Adapters for concentrator tubes.

6.17. Glass fiber filters, Whatman GF-D, GF-F, GMF150, or equivalent.

6.18. Solid phase extraction discs, 3M 90mm C18, or equivalent.

6.19. Dean-Stark trap, 5 or 10 mL, with T-joints, condenser and 125 mL flask.

6.20. Continuous liquid-liquid extractor.

6.21. All-glass Soxhlet apparatus, 500 mL flask.

6.22. Soxtherm extraction apparatus (or equivalent), including glass thimble holders, glass beakers, and gaskets.

6.23. Glass funnels, sized to hold 170 mL of liquid.

6.24. Desiccator.

6.25. Turbo evaporator

6.26. Rotary evaporator with a temperature controlled water bath.

6.27. High speed tissue homogenizer, equipped with an EN-8 probe or equivalent.

6.28. Glass wool, extracted with methylene chloride, dried and stored in a clean glass jar.

6.29. Vacuum extraction device for solid phase extraction, 1 Liter glass funnel with 90mm filter disc holder with a vacuum source, Kontes or equivalent.

7. REAGENTS AND STANDARDS

7.1. Column Chromatography Reagents

7.1.1. Silica Gel - Kieselgel 60 or equivalent, activate for 1 hour at 184°C before use. Store at 130°C in covered flask.

7.1.2. Acid Alumina - ICN or equivalent, activated as necessary.

7.1.3. Basic Alumina - ICN or equivalent. No activation required.

7.1.4. Granular carbon/silica gel - Mix 3.6 g granular carbon and 16.4 g activated silica gel; (alternatively, prepare carbon/silica gel (5%/95%); i.e., combine 5 g precleaned carbon with 95 g silica gel). Store at room temperature in a Teflon ® lined covered jar. The first LCS prepared with a new batch of column packing material is the quality control check of the packing materials. Refer to historical control limits before accepting the new batch of material.

7.1.5. 44% H_2SO_4 /silica gel - Mix 24 mL conc. H_2SO_4 and 56 g activated silica gel. Stir and shake until free flowing. Store at room temperature.

7.1.6. 33% NaOH/silica gel - Mix 34 mL 1N NaOH and 67 g activated silica gel. Stir and shake until free flowing. Store at room temperature.

7.2. Acid Alumina Activity Assessment

Alumina activity may vary with the matrix or environmental conditions. Monitor internal standard and cleanup recovery standard recoveries in extract analysis. Low recoveries of cleanup recovery standard (CRS) may indicate loss of alumina activity. Assess stability of alumina activity and apply corrective action as appropriate (reactivate and reprofile).

Note: a column profile should be done to show elution of all 2,3,7,8 substituted analogs so problems can be readily identified.

7.2.1. Profile each vendor lot of activated alumina as corrective action for low internal standard and CRS recoveries dictate. If necessary, proceed as follows:

7.2.1.1. Set up and label 3 acid alumina columns.

7.2.1.2. Pre-rinse with 20 mL hexane.

7.2.1.3. Add 2 mL hexane spiked with internal standards and natives (spike amounts equivalent to those for LCS) with 2X2 mL hexane rinse of fractions.

7.2.1.4. Elute each column with 20 mL hexane. Collect and label these fractions.

7.2.1.5. Elute each column with 5 x10 mL methylene chloride/hexane at the appropriate v/v percent. Collect and label these fractions separately.

7.2.1.6. Elute each column with 10 mL of 100% methylene chloride. Collect and label these fractions. Reduce all fractions to final volume and add recovery standard.

7.2.2. Review data and select an elution scheme. Group the fraction from each solvent system as follows:

7.2.2.1. Pre-analyte fraction - consists of all eluent prior to elution of first target analytes.

7.2.2.2. Analyte fraction - consists of all that contain detectable levels of target analytes.

7.2.2.3. Post-analyte fraction - consists of all eluents after elution of the last target analyte.

7.2.3. Select the solvent system which best meets the following two conditions:

7.2.3.1. Pre-analyte fraction consists of 20mL hexane and no more than 20 mL mixed solvent.

7.2.3.2. Analyte fraction consists of no more than 20mL of mixed solvent and contains greater than 90% of all target analytes and greater than 80% of all internal standards.

7.2.4. After selection of the appropriate solvent system and fractionation pattern, perform triplicate acid alumina cleanups on spiked hexane to ensure reproducibility of the fractionation pattern. Document each elution scheme.

7.2.5. Each subsequent batch of acid alumina used in the lab (from the same vendor lot) must be checked for stable activity.

7.3. Reagents

7.3.1. Sulfuric acid, concentrated, ACS grade, specific gravity 1.84.

7.3.2. Distilled water demonstrated to be free of interferents

7.3.3. 1 N HCl.

7.3.4. Silica gel.

7.3.5. Solution for breaking emulsions: Slowly add 1.0L of reagent grade NaOH solution to a 2.0L NaOH container, containing 1.0L of DI H2O, and leave the container in secondary containment with the lid off.

Warning: The solution will begin to heat so let the solution stand until equilibrium is met and the solution is at room temperature.

When this process is complete, the solution will then be ready for use in the samples.

7.3.6. Precleaned Sodium Sulfate.

7.3.7. Canola Oil (for tissue extraction only), or other suitable oil.

7.4. Desiccating Agent

7.4.1. Sodium sulfate, granular, anhydrous.

7.5. Solvents

7.5.1. High-purity, distilled-in-glass or highest available purity: Methylene chloride, hexane, methanol, tetradecane, isooctane, toluene, cyclohexane, and acetone.

7.6. All daily internal standard, daily clean up recovery standards, and daily spiking solutions are stable for one year from preparation. After 1 year, solutions may be re-verified. The re-verified solution may be used for an additional year, or until there is evidence of compound degradation or concentration. The re-verification must be performed using an unexpired, not previously re-verified solution from a second lot or second vendor.

7.6.1. Sealed ampules may be used until the manufacturer's expiration date is exceeded. If no expiration date is provided, then the expiration date will be 10 years from the date the ampule is opened. The solvent level should be monitored prior to each use to assure there has been no concentration of the standard over time.

7.6.2. Standards for method 8290A require storage at ≤ 6°C.

7.7. Field Surrogate Solution (air matrices)

This solution contains one ^{37}Cl labeled analog (for Method TO-9/TO-9A) or one ^{37}Cl and four ^{13}C labeled analogs (for Method 0023) at the nominal concentration indicated in Table 2. It is used to assess sample collection and recovery procedures.

7.8. Internal Standard

This isooctane solution contains the nine internal standards at the nominal concentrations that are listed in Table 2. The solution contains at least one carbon-labeled standard for each homologous series, and it is used to measure the concentrations of the native substances. (Note that $^{13}C_{12}$ -OCDF is not present in the solution.)

7.9. Native Spike Standard

Also known as the Matrix Spike or Native Spike solution. Contains all the 2,3,7,8-substituted unlabeled analytes listed in Table 2. Prepare using the appropriate standards to yield a spiking solution with a concentration of 4.0 ng/ml for the tetra-CDDs/CDFs, 20 ng/ml for the penta-, hexa-, and hepta- CDDs/CDFs, and 40 ng/ml for the octa- CDD/CDF.

7.10. Recovery Standard Solution

This tetradecane solution contains two recovery standards ($^{13}C_{12}$-1,2,3,4-TCDD and $^{13}C_{12}$ -1,2,3,7,8,HxCDD). An appropriate volume of this solution is spiked into each sample extract before the final concentration step.

7.11. Cleanup Recovery Standard Solution (CRS)

Prepare $^{37}Cl_4$-2,3,7,8-TCDD at the concentration shown in Table 2, in isooctane (or toluene).

7.12. Preparation and QC of PUF material

7.12.1. The PUF material is purchased pre-cut.

7.12.2. The PUFs are rinsed by Soxhlet with acetone (or other appropriate solvent) for a minimum of 16 hours and air dried for a minimum of 2 hours in a contaminant-free area.

7.12.3. One PUF from the rinsed batch is randomly selected to be the QC sample for the batch.

7.12.4. The PUF is loaded into a pre-cleaned Soxhlet extractor charged with toluene.

7.12.5. The 1613/8290 daily internal standard solution is spiked into the PUF and it

is extracted for a minimum of 16 hours.

7.12.6. The Soxhlet extract is recovered and processed according to Section 11.4.

7.12.7. The batch of PUF is considered acceptable if no target analytes are detected at or above the laboratory or project specific reporting limit.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. The sample collection, shipping, handling, and chain-of-custody procedures are not described in this document. Sample collection personnel will, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory. The analyst should make a judgment, based on the appearance of the sample, regarding the necessity for additional mixing. If the sample is clearly non-homogeneous, the entire contents should be transferred to a glass or stainless steel pan for mixing with a stainless steel spoon or spatula before removal of a sample portion for analysis.

8.2. Grab and composite samples must be collected in glass containers.

8.3. Ambient air samples are collected on a Quartz Fiber Filter followed by a glass sleeve containing a polyurethane foam plug.

8.4. Samples from stationary sources are collected on glass or quartz fiber filters and XAD-2 Resin. (See WS-ID-0009 for sample preparation procedures).

8.5. Conventional sampling practices must be followed. Do not rinse the bottle with sample before collection. Sampling equipment must be free of potential sources of contamination.

8.6. Grinding or blending of fish samples.

If not otherwise specified by the client, the whole fish (frozen) should be blended or ground to provide a homogeneous sample. The use of a stainless steel meat grinder with a 3 to 5 mm hole size inner plate is recommended. In some circumstances, analysis of fillet or specific organs of fish may be requested by the client. If so requested by the client, the above whole fish requirement is superseded. More detail can be found in "Tissue Sampling and Handling for a variety of Methods" (WS-WI-0018).

Warning: Hearing protection must be worn when grinding samples.

8.7. With the exception of the fish tissues, which must be stored at - 20°C, all samples should be stored at 4°C ± 2, extracted within 30 days and completely analyzed within 45 days of collection. The 30 day hold time is recommended. PCDDs and PCDFs have demonstrated stability for greater than one year.

8.8. All extracts must be stored capped, in the dark, at room temperature (approximately 21°C to 28°C). All extracts for method 8290A must be stored capped at ≤ 6°C.

8.9. For moisture determinations refer to SOP WS-OP-0013.

9. QUALITY CONTROL

9.1. One method blank (MB) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory matrix (reagent water, sodium sulfate, PUF, XAD, filter, etc.) processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. The method blank contains a PUF plug, XAD, or filter prepared from the same batch as the field samples whenever possible for air samples.

Certain programs, such as DOD, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the lower calibration limit.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.1.1. If the accompanying samples are aqueous, use distilled water as a matrix. Take the method blank through all steps detailed in the analytical procedure.

9.1.2. Use sodium sulfate as the method laboratory matrix when solids are extracted. Use a mixture of sodium sulfate and canola oil as the matrix when tissues are extracted. Take the method blank through all steps detailed in the analytical procedure.

9.1.3. The method blank must be spiked prior to extraction with the same amount of ^{13}C -labeled internal standards as added to samples.

9.1.4. If method blank contamination is present, check solvents, reagents, fortification solutions, apparatus and glassware to locate and eliminate the source of contamination before any further samples are extracted and analyzed. The presence of any analyte in the method blank ate concentrations greater than the reporting limit (RL) is cause for corrective action.

9.1.4.1. OCDD is a ubiquitous laboratory contaminant. A method blank and the associated samples are deemed acceptable if the OCDD

concentration is <5x the specified reporting limit. Flag data appropriately. The analyst is expected to investigate and eliminate potential sources of systematic contamination.

9.1.4.2. If a target analyte is detected in the blank but the associated samples are ND (not detected), then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.4.3. If a target analyte is detected in the blank, but the concentration of the contaminant in the samples >10x the blank concentration, then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.4.4. If one of the conditions above is not met then the sample associated with a contaminated method blank must be re-extracted.

9.1.5. If new batches of reagents or solvents contain interfering contaminants, purify or discard them.

9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, sodium sulfate, PUF, XAD, etc.) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See policy WS-PQA-003 for specific acceptance criteria. When associated with PUF samples, the LCS should contain a PUF plug prepared from the same batch as the field samples whenever possible.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.2.1. A LCS is deemed acceptable if control analytes are above control limits and the associated samples are ND, unless otherwise specified by the client. Note any actions in the narrative.

9.3. The assessment of matrix effects on method performance, as required by NELAP, is met in Method 8290 and 8290A, as in all isotope dilution techniques, with the use of isotopically labeled compounds. These isotopically labeled compounds are analogs of target analytes and are spiked into each sample. Therefore, matrix effects on method performance may be judged by the recovery of these analogs. Sample analysis

acceptance is controlled by the performance of these analogs in each sample. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair are extracted at the client's request only. Method 8290A does not address analysis of MS/MSD. An exception to this rule is a batch containing South Carolina samples for Method 8290. These batches must have an MS/MSD prepared. However, South Carolina requires Method 8290A after December 31, 2008. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. When requested by the client, the MS/MSD pair shall be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, the LCS, the selected field sample, and the MS/MSD may be required after evaluation and review. Matrix Spike/ Matrix Spike Duplicates are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. An LCS/LCSD may be extracted to show precision of the extraction and analysis process.

9.3.1. Matrix Spike (MS): A sample, which is spiked with a known amount of the matrix spike fortification solution prior to the extraction step. The recoveries of the matrix spike compounds are determined; they are used to estimate the effect of the sample matrix upon the analytical methodology.

9.3.2. Matrix Spike Duplicate (MSD): A second portion of the same sample as used in the matrix spike analysis and which is treated like the matrix spike sample.

9.3.3. Locate the sample for the MS and MSD analyses (the sample may be labeled "double volume").

9.3.4. Add an appropriate volume of the matrix spike fortification solution, adjusting the fortification level as specified in Table 1, under IS Spiking Levels.

9.3.5. The results obtained from the MS and MSD samples (percent recovery and concentrations of 2,3,7,8-substituted PCDDs/PCDFs) should agree within 20 percent relative difference. Report all results and flag outliers.

9.3.6. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.4. Duplicates

9.4.1. Upon client request, duplicates may be processed. Locate the sample specified for duplicate analysis, and prepare and analyze a second 10-g soil or sediment sample portion or 1 L water sample, or an appropriate amount of

the type of matrix under consideration. Duplicate samples are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. A duplicate injection of a sample extract may be performed to display instrument precision.

9.4.1.1. The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference. Report all results and flag outliers.

9.4.2. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.5. Field Blanks

9.5.1. Each batch of samples may contain a field blank sample of nominally uncontaminated soil, sediment or water that is to be processed for analysis.

9.5.1.1. Weigh a 10-g portion or use 1 L (for aqueous samples) of the specified field blank sample and add the appropriate amount of internal standard to yield 100 pg/µL in the final extract.

9.5.1.2. Extract by using the procedures described in Section 11. As applicable, add the appropriate amount of recovery standard to yield 100 pg/µL in the final extract. Analyze a 1-2 µL aliquot of the concentrated extract using SOP WS-ID-0005.

9.6. Rinsate Samples

9.6.1. In addition to the field blank, a batch of samples may include a rinsate, which is a portion of the solvent (usually trichloroethylene) that was used to rinse sampling equipment. The rinsate is analyzed to assure that the samples were not contaminated by the sampling equipment.

9.6.2. The rinsate sample must be processed like a regular sample.

Take a 100-mL (± 0.5 mL) portion of the sampling equipment rinse solvent (rinsate sample), filter, if necessary, and add the appropriate amount of internal standard to yield 100 pg/µL in the final extract.

9.6.3. Using appropriate methods, concentrate to approximately 10 mL.

9.6.4. Just before analysis, add the appropriate amount of recovery standard to yield 100 pg/µL in the final extract. Reduce the volume to a final volume of 20 µL, as necessary. No column chromatography is required.

9.6.5. Analyze an aliquot following the same procedures used to analyze samples.

9.7. Surrogate/Clean Up Recovery Standard

A surrogate compound may be spiked into all air media samples prior to collection. For all other matrices, a clean up recovery standard is spiked following extraction and just prior to cleanup, in order to monitor relative loss of internal standard during both extraction and cleanup.

9.8. Internal Standards

An internal standard is a ^{13}C -labeled analog of a PCDD/PCDF congener. Internal standards are added to all samples including method blanks and quality control samples before extraction, and they are used to quantitate the concentration of the analytes. Nine internal standards are used in this method. There is one for each of the dioxin and furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight. Additional internal standards may be added to act as retention time references, but they are not used for quantitation.

9.8.1. A 2000 pg aliquot of the internal standard mixture is added to all samples, regardless of sample size. As an example, for $^{13}C_{12}$ -2,3,7,8-TCDD, a 10-g soil sample requires the addition of 2000 pg of $^{13}C_{12}$ -2,3,7,8-TCDD to give the requisite fortification level.

9.8.2. Internal standards must be spiked into all samples, QC samples, and included in all calibrations.

9.8.3. For each sample and QC aliquot, calculate the percent recovery. The percent recovery should be between 40 percent and 135 percent for all nine internal standards.

9.8.4. A low or high percent recovery for a blank does not require discarding the analytical data but it may indicate a potential problem with future analytical data. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.9. Recovery Standard: Two recovery standards are used to determine the percent recoveries for the internal standards. The $^{13}C_{12}$ -1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated internal standards while $^{13}C_{12}$ -1,2,3,7,8,9-HxCDD is used to determine the recovery of the hexa-hepta- and octachlorinated internal standards. $^{13}C_{12}$ -1,2,3,7,8,9-HxCDD also acts as a retention time reference for the unlabeled analog present in sample extracts. They are added to the final sample extract before HRGC/HRMS instrument analysis.

9.10. Recommended Corrective Actions and Troubleshooting Steps

- Verify satisfactory instrument performance.
- If possible, verify that no error was made while weighing the sample aliquots.
- Review the analytical procedures with the performing laboratory personnel.

10. CALIBRATION

10.1. On a daily basis, calibrate any balance to be used in accordance with SOP WS-QA-0041.

10.2. On a monthly basis, calibrate any autopipettor to be used in accordance with SOP WS-QA-0004.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2. Refer to SOP WS-ID-0009 for the preparation of stationary source samples.

11.3. Sample Pre-Treatment

11.3.1. Paper Pulp Sludges are generally air-dried and ground prior to extraction following Section 11.5. Because of the drying procedure, a Dean-Stark water separator is optional for extraction.

11.3.2. Fly Ash — Fly ash samples are pretreated with HCl prior to extraction by both soxhlet and separatory funnel techniques.

11.3.2.1. Weigh 2-10g of sample aliquot into a clean glass jar.

11.3.2.2. Add 1.0mL of the internal standard mixture with 2 mL of acetone.

11.3.2.3. Add 150 mL of 1N hydrochloric acid and shake for 4 hours.

11.3.2.4. If the sample reacts violently with acid, then allow the sample to equilibrate for 4 hours with no shaking.

11.3.2.5. Filter the contents of the jar through a glass fiber filter.

11.3.2.6. Extract the solids as per Section 11.5, omitting the daily internal standard spike for the samples.

11.3.2.7. Extract the aqueous filtrate as per Section 11.8, using 100 mL of toluene for the first shake, and 100 mL of hexane for subsequent shakes.

11.3.2.8. Concentrate the combined toluene solutions to near dryness on a rotary evaporator at 50°C. Proceed with Section 11.12 as necessary.

Note: As an option, a Soxhlet/Dean Stark extractor system may be used, with toluene as the solvent. No sodium sulfate is added when using this option.

11.4. Waste Dilution (Still-Bottom/Fuel Oil, and other solvent-miscible materials).

11.4.1. Weigh 1 g of the waste (organic liquids, fuel oils, and solids that will dissolve in a solvent) into a vial.

11.4.2. Add 40 mL of toluene (or other solvent if the material is not miscible/soluble in toluene). Shake gently to dissolve.

11.4.3. Remove a 4.0 mL aliquot (0.1g sample equivalent) and place in a culture tube. Add 1.0 mL of daily internal standard and 1.0 mL of cleanup recovery standard, and proceed to Section 11.12.

11.5. Soxhlet Extraction (Solids, Tissues, Sludges, Wipes)

11.5.1. Pre-extract the glassware by heating the flask until the toluene is boiling. When properly adjusted, 1-2 drops of toluene per second will fall from the condenser tip into the receiver. Extract the apparatus for a minimum of four hours.

WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensors are cold before you turn the heating element on. Check all of the condensors about 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If this cleaning cycle is to be left unattended (e.g., overnight) the first chemist to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.

11.5.2. After pre-extraction, cool and disassemble the apparatus.

11.5.3. If tissues requiring % Lipids are to be extracted, for each sample weigh the concentration vessel with label and boiling chips. Record the mass on the benchsheet. Refer to SOP WS-QA-0018 "Subsampling", for instructions on how to homogenize and subsample the container of sample.

11.5.4. Weigh a well-mixed aliquot of each sample (10 g, unless otherwise specified) into a clean Soxhlet thimble. Record the mass to the nearest 0.01g. Use sodium sulfate for the batch QC (MB, LCS) for solids, and a mixture of 9 g sodium sulfate and 1 g canola oil for the batch QC for tissue matrices.

11.5.4.1. In the case of wipes, place the entire wipe sample into the Soxhlet apparatus (no thimble needed), including any liquid present with the sample. Use pre-cleaned wipes for the batch QC samples.

11.5.5. Place the thimble into a Soxhlet apparatus equipped with a Dean-Stark water separator.

11.5.6. Spike all samples with 1.0 mL of internal standard solution (2 pg/µL), for a final concentration of 200 pg/g (based on a 10 g sample).

11.5.7. Spike the LCS (and MS/MSD, if present) with 50 uL of native spike.

11.5.8. Reassemble the pre-extracted apparatus and add a fresh charge (250-300 mL) of toluene to the receiver and reflux flask.

11.5.9. Reflux 16 hours, with the solvent cycling at least 5 times per hour.

WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensors are cold before you turn the heating element on. Check all of the condensors about 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If this cleaning cycle is to be left unattended (e.g., overnight) the first chemist to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.

11.5.10. Drain the water from the receiver if the receiver fills with water. Check and drain when necessary.

Note: If the receiver holds 10 mL of liquid, and 20 g of an approximately 10% solid sample is being extracted, then approximately 9 mL of water will end up in the receiver. In this case, the receiver will not need to be emptied (insufficient liquid to overflow), but it should be checked. If the sample amount is 50, and the percent solids is still 10%, then 45 mL of water will end up in the receiver. In this case, frequent checking is required, and the receiver will need to be emptied at least 5 times.

11.5.11. After refluxing, allow the apparatus to cool.

11.5.12. If samples DO NOT require % lipids add 100 µL of tetradecane as a keeper to the round bottom flask.

11.5.13. Proceed to Section 11.17.

11.6. SoxTherm Extraction (Solids, Tissues, Sludges,Wipes)

11.6.1. Prior to loading samples, run the system through 2 cleaning cycles (approximately 1 hour each).

11.6.2. After pre-extraction, cool and disassemble the apparatus.

11.6.3. Weigh a well-mixed aliquot of each sample (10 g, unless otherwise specified) into a clean Soxhlet thimble. Record the mass to the nearest 0.01g. Use sodium sulfate for the batch QC (MB, LCS) for solids, and a mixture of 9 g sodium sulfate and 1 g canola oil for the batch QC for tissue matrices.

11.6.3.1. In the case of wipes, place the entire wipe sample into the Soxhlet apparatus (no thimble needed), including any liquid present with the sample. Use pre-cleaned wipes for the batch QC samples.

11.6.4. Place the thimble into the Soxtherm apparatus.

11.6.5. Spike all samples with 1.0 mL of internal standard solution (2 pg/µL), for a final concentration of 200 pg/g (based on a 10 g sample).

11.6.6. Spike the LCS (and MS/MSD, if present) with 50 uL of native spike.

11.6.7. Reassemble the pre-extracted apparatus and add a fresh charge (150 mL) of toluene to the apparatus.

11.6.8. Program the system to boil for 1 hour, and reduce the toluene volume by 70-90 mL (volume < volume of the thimble).

11.6.9. Continue the extraction for one hour fifteen minutes, reducing the toluene volume by another 15 mL.

11.6.10. After refluxing, allow the apparatus to cool.

11.6.11. Pour the samples into round bottom flasks, and if samples DO NOT require % lipids add 100 µL of tetradecane as a keeper to the round bottom flask.

11.6.12. Proceed to Section 11.17.

11.7. Extract Splitting (Wipes)

Wipe extracts prepared using either Soxhlet or shaking techniques are split prior to further workup, to permit an archive aliquot, or analysis by an additional method.

Once the extract has been concentrated using the rotovap or Turbovap, proceed as follows:

11.7.1. Add approximately 1 mL of hexane or toluene to rinse the sides of the round bottom flask. Using a pipette, withdraw the sample from the round bottom flask and transfer the liquid into a test-tube. Use additional amounts of solvents to rinse the flask. Transfer all the liquid into the test-tube. Ensure that all traces of sample in the round bottom flask have been thoroughly rinsed from all surfaces. Bring the sample volume to 8.0 mL or 10.0 mL (or appropriate volume) with the addition of rinse solvent.

11.7.2. Upon completion of the rinsing, cap the test tube and shake vigorously. Take ½ of each sample (or an appropriate amount as instructed by the client, program manager or department manager) and transfer to a culture tube. Archive the remaining sample for future use.

11.7.2.1. If only one analysis is required, then ½ of the sample is archived and the other half is analyzed.

11.7.2.2. If "N" analyses are required, then the extract is divided into "N+1" equal portions, so that one portion is archived, and a portion is used for each test.

11.8. Aqueous Samples (liquid/liquid extraction).

11.8.1. When setting up the glassware for a batch, for each sample label one separatory funnel and one 500 mL round-bottom flask with the sample ID.

11.8.2. Weigh the sample in the bottle on the top loading balance to the nearest centigram (0.01g), and record the mass.

11.8.3. For each sample, add 1 mL of daily internal standard solution into 2 mL of acetone. Add this solution to the sample in the separatory funnel. Each aliquot of spike mixture is added similarly.

11.8.4. Dissolve 50µL of the target analyte into acetone and add this mixture into the LCS container.

11.8.5. Pour the entire sample (approximately 1L) into a 2L separatory funnel that is labeled with the sample ID.

11.8.6. Add 100 mL methylene chloride to the sample bottle, seal, and shake for 30 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel.

11.8.7. Create a blank and LCS by adding 1 L of laboratory reagent water to 2

additional separatory funnels. Add 100 mL methylene choride to each funnel.

11.8.8. To the LCS, add 50 µL of the precision and recovery standard dissolved into 2 mL of acetone.

11.8.9. Extract the samples by shaking each funnel for two minutes with periodic venting.

Warning: Separatory funnel extraction with methylene chloride is a high-risk activity. Pressure may build rapidly in the funnel. It should be vented after several seconds of shaking, and often enough to prevent build-up of pressure. Chemist performing separatory funnel extraction must wear a face shield over their safety glasses/goggles. Alternatively, the extraction can be performed behind a closed fume hood sash.

11.8.10. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation.

11.8.11. Repeat the extraction two additional times with methylene chloride.

11.8.12. Determine the original sample volume by re-weighing the sample bottle. Record the sample volume to the nearest centigram (0.01g).

11.8.13. Dry extract with sodium sulfate: Place glass wool in a precleaned filter funnel. Rinse glass wool with methylene chloride and load funnel with Na_2SO_4. Pour extract through Na_2SO_4 to remove water. Rinse Na_2SO with fresh methylene chloride and collect in round bottom flask.

11.8.14. Transfer the extract to a 500 mL round-bottom previously labeled with the sample ID, then add approximately 100 µL of tetradecane and concentrate on a rotary evaporator or TurboVap.

11.8.15. Perform macro-concentration as detailed in Section 11.17.

11.9. Aqueous Samples (solid phase extraction).

11.9.1. Weigh the sample in the bottle on the top loading balance to the nearest centigram (0.01g), and record the mass.

11.9.2. Create a blank and LCS by adding 1L of laboratory reagent water to 2 additional 1L bottles.

11.9.3. For each sample, add 1mL of daily internal standard solution in acetone.

Add this solution to the sample in the bottles. Each aliquot of spike mixture is added similarly.

11.9.4. To the LCS, add 50µL of the precision and recovery standard in acetone.

11.9.5. Prepare the C18 extraction discs by first soaking them in toluene for at least 5 minutes.

11.9.6. Assemble the filter holder and vacuum filtration flask and place the extraction disc onto the filter holder. Place a GF-F filter on top of the extraction disc. If the sample has a large amount of particulates a GF-D filter can be placed on top of the GF-F filter. Alternatively, a GMF-150 filter can be used in place of the two filters.

11.9.7. Place the filtering funnel onto the disc holder and clamp it in place.

11.9.8. Rinse the filter and discs with approximately 15mL of toluene and allow it to soak for about a minute. Apply vacuum and draw the toluene through the discs. Repeat the wash step using about 15mL of acetone. Apply vacuum and draw the acetone through the discs.

11.9.9. Rinse the filter and discs with approximately 15mL of methanol and allow it to soak for about a minute. Apply vacuum and draw the methanol through the discs, but **DO NOT ALLOW THE DISCS TO GO DRY**. If they do go dry, simply repeat the methanol rinse step, leaving a 1 – 2mm layer of solvent on top of the discs.

11.9.10. Rinse twice with about 50mL of reagent water, leaving a 1 – 2mm layer of water on the surface of the discs.

11.9.11. Pour the spiked method blank, LCS or sample into the reservoir and apply vacuum to begin the extraction. Adjust the vacuum such that the extraction takes approximately 10 minutes. Samples with large amounts of particulates may take much longer.

11.9.12. After most of the sample has been pulled through the discs, rinse the sample bottle with a few mLs of reagent water and add the rinse to the funnel. Rinse down the sides of the funnel with reagent water as well.

11.9.13. Allow the discs to dry, remove them from the holder and extract by soxhlet (11.5) or soxtherm (11.6) and proceed with cleanups.

11.9.14. Determine the original sample volume by re-weighing the sample bottle. Record the sample volume to the nearest centigram (0.01g).

11.10. Breaking Emulsions

There are several useful methods to decrease or eliminate emulsion in aqueous samples when extracting with methylene chloride. These methods may include stirring with a pipette to manually breakup the emulsions or to transfer the sample into centrifuge tubes and centrifuge at approximately 3000 RPM. The most useful method is to use a 10:1 $NaOH/H_2O$ solution to change the pH enough to disrupt the emulsion phase, which works 90% of the time. See Section 7.3.5 for reagent preparation.

11.10.1. Check the pH of the sample to verify that the pH is between 3 and 7. If the pH is greater than 7, consult the supervisor and client for instructions.

11.10.2. Pour approximately 100 mL of the 10:1 $NaOH/H_2O$ into a 1 L amber glass bottle (AGB).

11.10.3. Drain the sample with the emulsion from the 2 L separatory funnel into the 1 L AGB and let it stand.

11.10.4. Empty the aqueous waste into the LLE waste drum.

11.10.5. Pour the solution with methylene chloride back into the same 2 L separatory funnel and drain the methylene chloride phase through Na_2SO_4 into a 500 mL round-bottom flask.

11.10.6. Empty the aqueous waste into the LLE waste drum.

11.10.7. Proceed with macro-concentration (Section 11.17).

11.11. Filter/PUF Samples

11.11.1. Place the glass sleeve containing the PUF and the Quartz Fiber Filter into the pre-cleaned Soxhlet extractor charged with toluene.

11.11.2. Add 2 mL (4000 pg) of 1613/8290 daily Internal Standard solution to all samples and QC.

11.11.3. Add 50 uL of 1613/8290 Native Spike to the LCS.

11.11.4. Extract the samples and QC for a minimum of 16 hours.

11.11.5. Concentrate the extract from the round bottom flask with hexane and adjust the volume.

11.11.6. Transfer the extract from the round bottom flask with hexane and adjust the volume.

11.11.7. Split the extract 50:50 for analysis and archive.

11.11.8. Proceed to Section 11.12.

11.12. Extract Clean-Up

11.12.1. For all samples that are not air media, spike 1.0 mL of the Cleanup Recovery Standard (CRS) prior to any cleanup into the round bottom flasks containing the samples and QC Extracts (See also Section 9.7).

11.12.2. Proceed with further cleanups as dictated by the sample matrix and extract color. The "Option C" cleanup (Section 11.13) and the IFB Upper Column cleanup (Section 11.14) are applied to samples with high levels of interferences. The IFB column cleanup (Section 11.15) is applied to all samples.

11.13. Acid Partitioning ("Option C")

11.13.1. Use this clean up as needed on samples with high levels of interferences. Consult with a lead chemist or department manager to determine applicability.

11.13.2. Partition the extract in 50-125 mL of hexane against 40 mL concentrated H_2SO_4 in a separatory funnel. Shake for two minutes. Remove and discard the H_2SO_4 layer (bottom). Repeat the acid washing until no color is visible in the acid layer (perform a maximum of four acid washings).

Warning: Shaking with a concentrated caustic is a high-risk activity. Analyst must wear a face shield over safety glasses/goggles, or the shaking must take behind a closed hood sash.

11.13.3. Partition the extract against 50 mL of distilled H_2O. Shake for two minutes. Remove and discard the aqueous layer (bottom). Dry the extract by pouring it through a funnel containing anhydrous sodium sulfate and collect it in a round-bottom flask. Rinse the sodium sulfate with two 15 mL portions of hexane, add the rinsates to the flask, and concentrate the hexane solution to near dryness on a rotary evaporator (35°C water bath), making sure all traces of toluene (when applicable) are removed. (Use of blow-down with an inert gas to concentrate the extract is also permitted.) The DI H_20 partition is applied only as samples warrant it at the discretion of the analyst.

11.14. IFB Upper Column Cleanup

11.14.1. Use this clean up as needed on samples with high levels of interferences. Consult with a lead chemist or department manager to determine applicability.

11.14.2. Set up the upper of the two chromatography columns as depicted in Figure 2.

The column (20 mm diameter) is packed in this order: a glass wool plug, 2 g activated silica gel, 4 g Acid silica gel, 2 g activated silica gel, and 1 g sodium sulfate.

11.14.3. Pre-rinse the column with 20 mL hexane, and discard the rinsate.

11.14.4. Add extract to the column. Rinse extract vessel 2 times with 1 mL each of hexane and add to column.

11.14.5. Elute 60 mL hexane directly onto acid silica column (upper column).

11.14.6. Collect the eluate, and concentrate before proceeding with the IFB cleanup (Section 11.15).

11.15. IFB Column Cleanup

Most samples will undergo this cleanup, either direction following concentration on the rotovap, or following the cleanup in Section 11.13 (Option C) or Section 11.14 (IFB Upper Column).

11.15.1. Set up two chromatography columns as depicted in Figure 2. The upper column (20 mm diameter) is packed in this order: a glass wool plug, 2 g activated silica gel, 4 g Acid silica gel, 2 g activated silica gel, and 1 g sodium sulfate. The lower column (15 mm diameter) is packed in this order: a glass wool plug, 6 g acid alumina, and 1 g sodium sulfate.

11.15.2. Pre-rinse each column with 20 mL hexane, and discard the rinsate.

11.15.3. Put one column above the other.

11.15.4. Add extract to the top column (silica column). Rinse extract vessel 2 times with 1 mL each of hexane and add to column.

11.15.5. Elute 60 mL hexane directly onto acid silica column (upper column).

11.15.6. Discard upper column.

11.15.7. Elute lower column with 10 mL of 20% methylene chloride/hexane. Discard in proper waste stream.

11.15.8. Elute lower column with 30 mL of 65% methylene chloride/hexane. Save and collect in culture tube.

11.15.9. Proceed with additional cleanups as necessary.

11.16. Carbon Column Clean-up (D2 Column)

Prepare an activated Carbon & Silica Gel column as described in below. Refer to the diagram in Figure 3 as well.

11.16.1. Push a glasswool plug down to the 3 inch mark in a pre-cut D2 column.

11.16.2. Add 1 g of 5% activated carbon/silica. Top with a glasswool plug.

11.16.3. With the column oriented with "A" on the top (and the carbon on the lower end of the column), pre-elute with 5 mL 1:1 methylene chloride :cyclohexane.

11.16.4. Discard pre-eluates.

11.16.5. Invert the column so that the column is oriented with the "B" on the top and pre-elute with 3 mL of 1:1 methylene chloride.

11.16.6. Dilute the extract to 1 mL with hexane and transfer to the column (still oriented in the "B" direction).

11.16.7. Rinse sample vial onto the column with 2 x 2 mL 1:1 methylene chloride:cyclohexane.

11.16.8. Elute with 6 mL 1:1 methylene chloride :cyclohexane

11.16.9. Elute with 5 mL 75:25 methylene chloride:methanol

11.16.10. Discard eluates.

11.16.11. Turn the column over (so that the "A" end is on top), and elute with 30 mL of toluene. Collect this eluate.

11.16.12. Concentrate to NEAR dryness using the Rotovap (Section 11.17) or Turbovap (Section 11.18), then proceed to the recovery standard step (Section 11.19).

11.17. Macro-concentration (Rotary Evaporator)

Concentrate the extracts in separate round bottom flasks on rotary evaporator.

11.17.1. Assemble the rotary evaporator according to manufacture's instructions, and warm the water bath. On a daily basis, preclean the rotary evaporator by solvent rinsing. Between samples, 2-3 mL rinses of toluene followed by a 2-3 mL rinse of hexane should be rinsed down the feed tube into a waste beaker.

Rotovap Conditions		
Solvent	**Bath Temperature (C)**	**Vacuum Setting (PSI)**
Toluene	80	25
Hexane	65	15
Methylene Chloride	70	No vacuum applied

11.17.2. Attach the round bottom flask containing the sample extract to the rotary evaporator. Slowly apply vacuum to the system, and begin rotating the sample flask.

11.17.3. Lower the flask into the water bath and adjust the speed of rotation and the temperature as required. At the proper rate of concentration, the flow of solvent into the receiving flask will be steady, but no bumping or visible boiling of the extract will occur.

NOTE: If the rate of concentration is too fast, analyte loss may occur.

11.17.4. For samples requiring % Lipids analysis:

11.17.4.1. Concentrate until the toluene has been completely removed. Add approximately 25 mL hexane and concentrate to ensure that only the lipids are present.

11.17.4.2. Dry the concentration vessel and let stand at room temperature. Weigh the vessel and record on the benchsheet.

11.17.4.3. Calculate % lipids as follows:

$$\%\,\text{Lipids} = \frac{\text{Final Vessel Mass - Initial Vessel Mass}}{\text{Sample Size}} \times 100\%$$

11.17.5. Proceed to extract cleanups, or transfer to a micro concentration vial for the recovery standard step (Section 11.19).

11.18. Micro-concentration (Turbovap)

Concentrate the extracts in 35 mL culture tubes in a turbo-evaporator. The turbo-evaporator model that the laboratory uses can hold up to 50-35 mL culture tubes. Other turbo-evaporator models can be used that may or may not have the same culture tube sizes and/or capacity. Adjust temperature according to solvent (65ºC for toluene and 45ºC for hexane or hexane/ methylene chloride mixtures)

11.18.1. The evaporating times are dependent on sample volume and solvent. The following are examples and can change from sample to sample. Each sample should be checked in intermittent intervals to make sure samples do not go dry.

11.18.2. When evaporating 30 mL toluene, it will normally take approximately 30-50

minutes with the temperature setting described above.

11.18.3. When evaporating 30 mL hexane/ methylene chloride, it will normally take approximately 20-30 minutes with the temperature setting described above.

11.18.4. For samples requiring % Lipids analysis refer to Section 11.17.4.

11.18.5. Proceed to extract cleanups, or transfer to a micro concentration vial for the recovery standard step (Section 11.19).

11.19. Recovery Standard

11.19.1. Transfer extracts to a micro concentration vial (test tubes and other small vessels may also be used)

11.19.2. With a stream of dry, purified nitrogen, reduce the extract volume to approximately 100 µL.

11.19.3. Add 20 µL of the recovery standard solution (Table 2).

11.19.4. With a stream of dry, purified nitrogen, reduce the extract volume to 20 µL.

11.19.5. Transfer the extract to an autoinjection vial and store in the dark at room temperature.

11.19.6. A smaller final volume can be used to decrease the detection limit upon client approval.

11.19.7. A larger final volume can be use to decrease potential matrix interferences, if the column and acid cleanups were unsuccessful.

11.20. Sample Dilution Procedure

11.20.1. Simple dilutions: Dilutions from 2X to 50X can be achieved without respiking the final extract. The calculation to determine the final extract concentration is as follows:

$$\text{Final Conc. of Extract} = \frac{(\text{Conc. of original extract}) \text{ x } (\text{Amount of aliquot taken})}{(\text{Volume of diluted extract})}$$

Ex:
$$\frac{(10\,\text{g}) \text{ x } (2\,\mu\text{L})}{(20\mu\text{L}) \text{ x } (100\mu\text{L})} = \frac{1\,\text{g}}{100\mu\text{L}}\ \text{FV}$$

Record the final sample concentration on the extract label.

11.20.2. Complex dilution requiring respiking of IS and RS:

Dilutions greater than 50x must be done by diluting and respiking the extract with IS and RS. This procedure may require serial dilution to be performed. If this procedure is done, then the sample size must be adjusted to reflect the aliquot taken.

Ex. 100X dilution (original sample with 10 g/20 µL final volume)

Take a 2 µL aliquot (1/10 of original sample) and add 18 µL of solvent keeper. Take a 2 µL aliquot of the dilution (1/100 of the original sample), respike with 1 mL IS and 20 µL RS, reduced to 20 µL FV.

Record the final sample concentration of the extract label.

12. CALCULATIONS/DATA REDUCTION

12.1. Not applicable

13. METHOD PERFORMANCE

It must be documented that all applicable system performance criteria specified were met before analysis of any sample is performed. .

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.1. The use of Roto-vaps and Turbo-vaps rather than Kuderna-Danish reduction allows extraction solvents to be collected and disposed of rather than released to the atmosphere.

14.2. Toluene, which is a less hazardous solvent, has been substituted for benzene as an extraction solvent.

14.3. The use of SoxTherm extraction rather than soxhlet extraction, when appropriate, reduces the volume of solvent used.

14.4. Standards should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards that must be discarded.

14.5. All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

14.6. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless they are being filled.

14.7. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Extracted aqueous/leachate samples contaminated with methylene chloride are collected at the fume hood in a 5-gallon or smaller carboy. If the samples are not at a

neutral pH, add small quantities of sodium bicarbonate to bring the waste to neutral. Stir well. Once neutralized, immediately pour the carboy contents into a blue plastic LLE drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the LLE drum to the waste collection area for shipment.

15.2. Extracted soil samples and thimbles, extracted PUF filters, XAD-2 resin, paper funnel filters, glass wool, sodium sulfate, assorted disposable glassware, fish/crawfish or similar materials, silica gel, alumina, and carbon from column clean-ups, contaminated with various solvents and eluates. Dump the materials into a orange contaminated lab trash bucket. When the bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Flammable solvent and methylene chloride waste generated during glassware and sodium sulfate cleaning. Solvent waste collected during roto-vap/turbo-vap reduction of extracted samples. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel solvent drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

15.4. Assorted flammable solvents and methylene chloride waste generated during quartz fiber filter preparation, PUF adsorbent preparation, XAD-2 resin preparation, PUF/XAD-2 cartridge preparation, glassware rinsing and sodium sulfate pre-rinsing.. Waste solvents and methylene chloride collected during roto-rap/turbo-vap reduction of extracted samples. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel drum in the H3 closet. When the drum is full to between two and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

15.5. Contaminated sulfuric acid used during extract cleanup. Collect the used sulfuric acid in empty, 2.5-liter, plastic coated jars. When full or after one year, whichever comes first, transfer these jars to the waste collection area for shipment.

15.6. Contaminated distilled water used during extract cleanup. Collect the contaminated water in a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the plastic LLE drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the plastic drum to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update IV. Method 8290A Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry February 2007.

16.2. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 8290 Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry September 1994.

16.3. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 0023A, Sampling Method for Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans Emissions from Stationary Sources. December 1996.

16.4. Compendium Method TO-9A "Determination of Polychlorinated, Polybrominated, and Brominated, Cholorinated Dibenxo-p-dioxins and Dibenzofurans in Ambient Air", EPA compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, second edition, January 1997.

16.5. Protocol for the Analysis of 2,3,7,8-TCDD by HRGC/HRMS". J. S. Stanley and T. M. Sack, EPA 600/4-86-004.

16.6. "Safety in Academic Chemistry Laboratories", American Chemical Society Publication, Committee on Chemical Safety (3rd Edition, 1979.)

16.7. "Carcinogens - Working with Carcinogens". Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control. National Institute for Occupational Safety and Health. Publication No. 77-206, August 1977.

16.8. "OSHA Safety and Health Standards, General Industry", (29 CFR 1910) Occupational Safety and Health Administration, OSHA 2206 (revised January 1976).

17. METHOD MODIFICATIONS

17.1. Deviations from EPA 8290 and 8290A.

17.1.1. Tetradecane instead of nonane is used as the final solvent to increase the stability of extracts and standards. Tetradecane is less volatile than nonane. Loss of analyte as a result of solvent incompatibility is monitored through recovery checks and calibration acceptance criteria.

17.1.2. Extract clean-ups are performed at the discretion of the analyst when interferences are observed. Then, the analyst should select the clean-up procedure appropriate to the interferent.

17.1.3. Section 7.4.6.4 of Method 8290 indicates that extracts should be transferred with hexane, then toluene. Toluene is used to transfer extracts to maintain compound solubility and minimize analyte loss.

17.1.4. Section 7.5.1.2 of Method 8290 specifies that a NaCl solution should be used for partitioning. Instead, the laboratory uses laboratory water only. NaCl is used to break up emulsions that may form. An analyst may use NaCl, NaOH, or any mechanical means to break up an emulsion.

17.1.5. Section 7.5.3 of Method 8290 specifies that hexane is used as a column elution solvent. The laboratory uses cyclohexane to achieve better and more reproducible separation of the target analyte from the interferent.

17.1.6. Carbon columns are packed with silica gel in place of celite. Elution solvents are changed accordingly. (SOP Section 11.4; Method 8290 Section 7.5.3.2, 8290A Section 7.3.6.).

17.2. Modifications from TO-9A method

17.2.1. Quartz Fiber Filters are cleaned by Soxhlet extraction with methylene chloride, not baked at 400 degrees C for 5 hours.

17.2.2. The PUF material may be pre-cleaned with methylene chloride or other appropriate solvent. The PUFs are not reused.

17.2.3. The $^{37}Cl_4$-2,3,7,8-TCDD surrogate is present at varying levels in the calibration curve (0.5-200 pg/ µL).

17.2.4. Samples are extracted with toluene not benzene.

17.2.5. Concentration is performed by rotary evaporation not Kuderna-Danish.

17.2.6. All cleanup procedures are optional and applied based on the analyst's discretion.

17.2.7. The laboratory uses 2 labeled recovery standard for the quantitation of labeled internal standards.

17.2.8. The final volume is adjusted to 20 µL in tetradecane.

17.2.9. Calibration and quantitation are performed in accordance to this SOP.

18. ATTACHMENTS

18.1. Table 1 - Types of Matrices

18.2. Table 2 - Composition of Sample Fortification and Recovery Standard Solutions.

18.3. Table 3 - The Seventeen 2,3,7,8-Substituted PCDD and PCDF Congeners

18.4. Figure 1 - Analysis Flowchart

18.5. Figure 2 — IFB column cleanup

18.6. Figure 3 — D2 Column cleanup

18.7. Appendix A - Periodic Wipe Test Performance

19. REVISION HISTORY

19.1. WS-IDP-0005, Revision 1.5, Effective 12/21/2012

19.1.1. Clarified extraction procedure by revising Section(s) 11.8.1- 11.8.4 and adding an extra extraction step (Section 11.8.3).

19.1.2. Editorial revisions. .

19.2. WS-IDP-0005, Revision 1.4, Effective 03/20/2012

19.2.1. Appended to Section 2.2: "This method can also use solid phase extraction (SPE), however, Test America West Sacramento is in the developmental stages for this extraction type and is not currently certified for its use."

19.2.2. Editorial changes.

19.3. WS-IDP-0005, Revision 1.3., Effective 06/10/2011

19.3.1. Added Section 11.9: Aqueous Samples (Solid Phase Extraction).

19.3.2. Editorial revisions.

19.4. WS-IDP-0005, Revision 1.2, Effective 2/11/2011

19.4.1. Added benzene to Section 5.2 Table..

19.4.2. Editorial revisions.

19.5. WS-IDP-0005, Revision 1.1, Effective 2/12/2010

19.5.1. Section 11.2 – updated SOP reference from SAC-ID-0009 to WS-ID-0009.

19.5.2. Section 11.6.1 – changed: "Prior to loading samples, run the system through

a cleaning cycle (approximately 3 hours)" to "(approximately 1 hour)."

19.5.3. Section 11.6.8 – changed "…fresh charge (140 mL) of toluene…" to "…fresh charge (150 mL) of toluene….".

19.5.4. Section 11.16.1 – inserted in Table "No vacuum applied" under vacuum setting (PSI) for solvent Methylene chloride.

19.6. WS-IDP-0005, Revision 1, Effective 10/2/2008

19.6.1. Added 8290A references.

19.6.1.1. Extract and standard storage.

19.6.1.2. Removal of MS/MSD.

19.6.2. Updated to TestAmerica format.

19.6.3. Separated the analytical steps from the preparation steps, this SOP is concerned only with the sample preparation.

19.7. WS-ID-0005, Revision 6.7, Effective 8/21/2008

19.7.1. Changed the word "toluene" to "acetone" in 7.11.2.

19.8. WS-ID-0005, Revision 6.6, Effective 4/9/2008

19.8.1. Added South Carolina rule to prepare an MS/MSD with every batch.

19.8.2. Modified to include extraction and analysis of ambient air samples collected in filter/PUF material.

TABLE 1

Types of Matrices, Sample Sizes and 2,3,7,8-TCDD-Based Method Calibration Limits (Parts per Trillion)

	Water	Soil Sediment Paper Pulp	Fly Ash	Human/ Fish Tissue	Adipose Tissue	Sludges, Fuel Oil	Still-Bottom	Ambient or Source Samples
Lower MCL(a)	0.01	1.0	2.0	1.0	2.0	10	20	40
Upper MCL(a)	4.0	400	400	400	400	2000	4000	8000
Weight (g)	1000	10	10	10	10	2.0	1.0	1 sample
IS Spiking Levels (ng)	2.0	2.0	2.0	2.0	2.0	2.0	2.0	4.0
Final Extract Volume (μL)	20	20	20	20	20	20	20	20

(a) For other congeners, multiply the values by 1 for TCDF, by 5 for PeCDD/PeCDF/HxCDD/HxCDF/HpCDD/HpCDF, and by 10 for OCDD/OCDF.

TABLE 2

Composition of the Sample Fortification and Recovery Standard Solutions

Analyte	Semple Fortification Solution Concentration pg/μL; Solvent: Isooctane	Recovery Standard Solution Concentration pg/μL; Solvent: Tetradecane
$^{13}C_{12}$ -2,3,7,8-TCDD	2[(a)], 100[(c)]	--
$^{13}C_{12}$ -2,3,7,8-TCDF	2[(a)], 100[(c)]	--
$^{13}C_{12}$ -1,2,3,4-TCDD	--	100
$^{13}C_{12}$ -1,2,3,7,8-PeCDD	2[(a)], 100[(c)]	--
$^{113}C_{12}$ -1,2,3,7,8-PeCDF	2[(a)], 100[(c)]	--
$^{13}C_{12}$ -1,2,3,6,7,8-HxCDD	2[(a)], 100[(c)]	--
$^{13}C_{12}$ -1,2,3,4,7,8-HxCDF[(d)]	2[(a)], 100[(c)]	--
$^{113}C_{12}$ -1,2,3,7,8,9-HxCDD	--	100
$^{13}C_{12}$-2,3,7,8-TCDD[(b)(c)]	0.8[(b)],100[(c)]	
	100[(c)]	
$^{13}C_{12}$ -2,3,4,7,8-PeCDF[(c)]	100[(c)]	
$^{13}C_{12}$ -1,2,3,6,7,8-HxCDF[(c)(d)]	100[(c)]	
$^{13}C_{12}$ -1,2,3,4,7,8-HxCDD[(c)]	100[(c)]	
$^{13}C_{12}$ -1,2,3,4,7,8,9-HpCDD[(c)]	100[(c)]	
$^{13}C_{12}$ -1,2,3,4,6,7,8-HpCDD	2[(a)], 100[(c)]	--
$^{13}C_{12}$ -1,2,3,4,6,7,8-HpCDF	2[(a)], 100[(c)]	--
$^{13}C_{12}$ -OCDD	4[(a)], 200[(c)]	--

(a) Standard 8290, Method 23, Method 0023A, TO9 and TO9A Sample Fortification Solution concentrations
(b) Method TO9 and TO9A surrogate concentrations
(c) Method 23 and Method 0023A surrogate concentrations
(d) $^{13}C_{12}$ -1,2,3,6,7,8-HxCDF is used as a Sample Fortification Solution and $^{13}C_{12}$-1,2,3,4,7,8-HxCDF is used as a surrogate solution in Method 23 and Method 0023A

TABLE 3

The Seventeen 2,3,7,8-Substituted PCDD and PCDF Congeners

PCDD	PCDF
2,3,7,8-TCDD(*)	2,3,7,8-TCDF(*)
1,2,3,7,8-PeCDD(*)	1,2,3,7,8-PeCDD(*)
1,2,3,6,7,8-HxCDD(*)	2,3,4,7,8-PeCDF
1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDD(+)	1,2,3,7,8,9-HxCDF
1,2,3,4,6,7,8-HpCDD(*)	1,2,3,4,7,8-HxCDF(*)
1,2,3,4,5,6,7,8-OCDD(*)	2,3,4,6,7,8-HxCDF
	1,2,3,4,6,7,8-HpCDF(*)
	1,2,3,4,7,8,9-HpCDF
	1,2,3,4,5,6,7,8-OCDF

(*)The ^{13}C -labeled analog is used as an internal standard.
(+)The ^{13}C -labeled analog is used as a recovery standard.

FIGURE 1 — Flowchart of Process

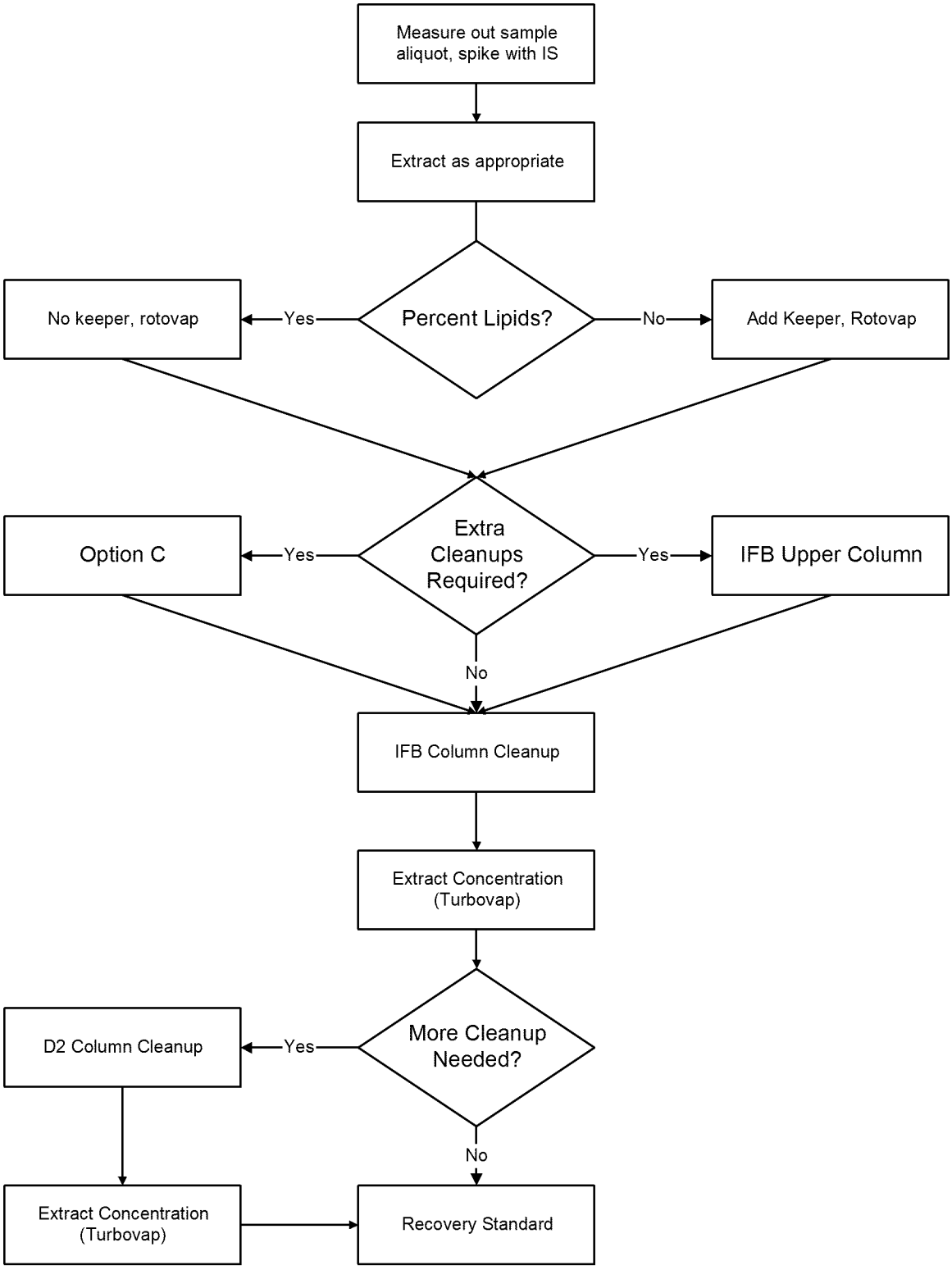


Figure 2 – Diagram of IFB Column Cleanup

Use 20 mm column for top column (IFB Column)
Use 16 mm column for bottom column* (Acid Alumina)
Note: Upper and lower columns are piggy backed for IFB cleanup, upper column only can be used for additional cleaning.

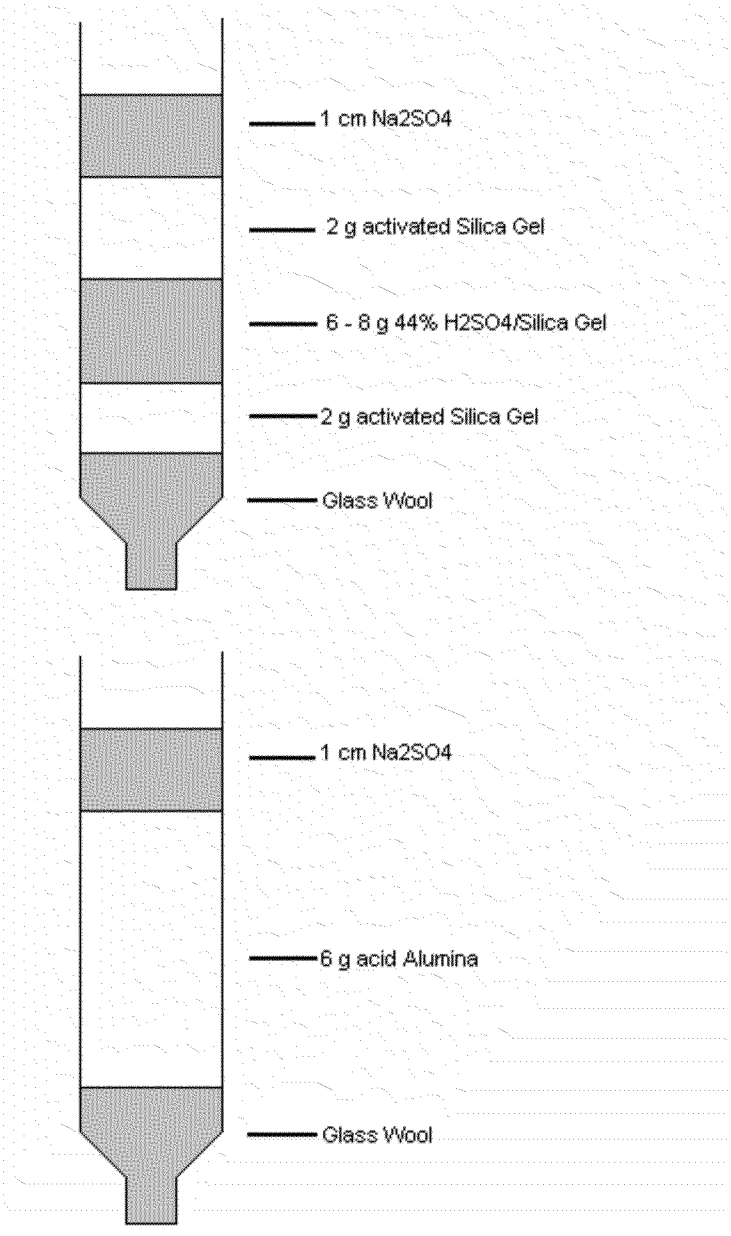
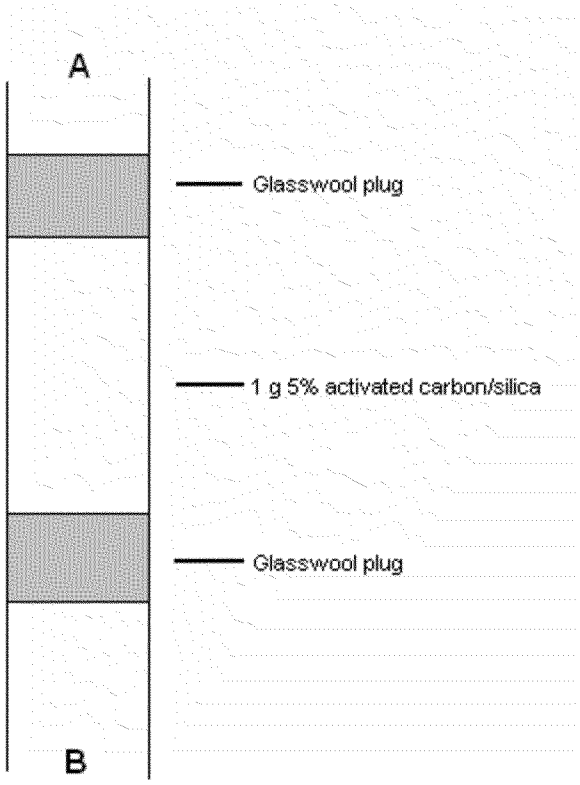


Figure 3— D2 Carbon Column:



APPENDIX A — Screening the Laboratory for 2,3,7,8 Congeners

This procedure is designed for the periodic evaluation of potential contamination by 2,3,7,8-substituted PCDD/PCDF congeners of the working areas inside the laboratory.

PERFORMING WIPE TEST
Perform the wipe tests on surface areas of two inches by one foot with laboratory wipers saturated with distilled-in-glass acetone or appropriate solvent using a pair of clean stainless steel forceps. Use one wiper for each of the designated areas. Combine the wipers to one composite sample in an extraction jar containing 200 mL distilled-in-glass hexane. Place an equal number of unused wipers in 200 mL hexane and use this as a control.

SAMPLE PREPARATION
Close the jar containing the wipes and 200 mL hexane and extract for 20 minutes using a wrist-action shaker. Use an appropriate means to reduce the volume to approximately 1.0 mL. Put through an alumina column to clean up potential interfering compounds. Add appropriate amount of recovery standard.

EXTRACT ANALYSIS
Concentrate the contents of the vial to a final volume of 20 µL (either in a minivial or in a capillary tube). Inject 2 µL of each extract (wipe and control) onto a capillary column and analyze for 2,3,7,8-substituted PCDDs/PCDFs as specified in the analytical method Section 11 (this exhibit). Perform calculations according to Section 12 (this exhibit).

REPORTING FORMAT
Report the presence of 2,3,7,8-substituted PCDDs and PCDFs as a quantity (pg or ng) per wipe test experiment (WTE). Under the conditions outlined in this analytical protocol, a lower limit of calibration of 25 pg/WTE is expected for 2,3,7,8-TCDD. A positive response for the blank (control) is defined as a signal in the TCDD retention time window at any of the masses monitored which is equivalent to or above 8 pg of 2,3,7,8-TCDD per WTE. For other congeners, use the multiplication factors listed in Table 1, footnote (a) (e.g., for OCDD, the lower MCL is 25 x 5 = 125 pg/WTE and the positive response for the blank would be 8 x 5 = 40 pg). Also, report the recoveries of the internal standards during the simplified cleanup procedure.

FREQUENCY OF WIPE TESTS
Wipe tests should be performed when there is evidence of contamination in the method blanks.

CORRECTIVE ACTION
An upper limit of 25 pg per TCDD isomer and per wipe test experiment is allowed. (Use multiplication factors listed in footnote (a) from Table 1 for other congeners.) This value corresponds to the lower calibration limit of the analytical method. Steps to correct the contamination must be taken whenever these levels are exceeded. To that effect, first vacuum the working places (hoods, benches, sink) using a vacuum cleaner equipped with a high-efficiency

particulate absorbent (HEPA) filter and then wash with a detergent. A new set of wipes should be analyzed before anyone is allowed to work in the dioxin area of the laboratory.

The test results and the decontamination procedure must be reviewed with EH&S.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Canton

SOP No. NC-OP-031, Rev. 6
Effective Date: 7/29/13

Title: EXTRACTION PROCEDURE FOR CHLORINATED ACID HERBICIDES BASED ON METHOD 8151A

[Method: SW846 Method 8151A]

Approvals (Signature/Date):

Technology Specialist — Date: 07/25/13	Health & Safety Coordinator — Date: 07/24/13
Quality Assurance Manager — Date: 07/24/13	Laboratory Director — Date: 07/28/13

This SOP was previously identified as SOP No. NC-OP-031, Rev 5-A, dated 05/28/12

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1. SCOPE AND APPLICATION

1.1 This method is applicable to the extraction of chlorinated herbicides in waters, solids, oils, and TCLP extracts. Appropriate compounds for extraction by this method are listed in SOP NC-GC-038.

2. SUMMARY OF METHOD

2.1 This method is based on SW846 Method 8151A. Aqueous samples are hydrolyzed if esters and acids are to be determined, then washed with methylene chloride by a separatory funnel extraction. After acidifying the sample, the free acids are extracted into diethyl ether.

2.2 Solids are extracted into methylene chloride/acetone by sonication. If esters and acids are to be determined, the extract is hydrolyzed and extracted into diethyl ether.

2.3 For both soils and aqueous samples, the free acid herbicides in the ether extract are esterified. The final volume is adjusted to prepare the extract for gas chromatography.

3 DEFINITIONS

3.1 Refer to the Test America Canton Quality Assurance Manual (QAM), current version, for definitions of terms and acronyms used in this document.

4 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. All glassware is cleaned per SOP NC-QA-014. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2 Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

4.3 Organic acids, especially chlorinated acids, cause the most direct interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure. The determination using pentafluorobenzylation is more sensitive, and more prone to interferences from the presence of organic acids or phenols than by methylation.

4.4 Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis. However, hydrolysis may result in the loss of dinoseb and the

formation of aldol condensation products if any residual acetone remains from the extraction of solids.

4.5 Sodium sulfate must be acidified.

4.6 Sample extracts must be dry prior to methylation, or poor recoveries will be obtained.

5. SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2 (Trimethylsilyl)diazomethane is an extremely toxic gas with an explosion potential. Since the explosion potential is catalyzed by imperfections in glass, generation of (Trimethylsilyl)diazomethane must be carried out in glassware free of scratches, cracks, chips, and that glassware which does not have ground glass joints. Solutions of (Trimethylsilyl)diazomethane will be kept at temperatures below 90°C. (Trimethylsilyl)diazomethane must be generated and handled in a fume hood.

5.3 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3 2 mg/m^3-Ceiling	This material will cause burns if comes into contact with the skin or eyes. Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision–even blindness.

Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain, and severe tissue burns. Can cause blindness.
Ethyl Ether	Flammable Irritant Peroxide Former	400 ppm-TWA	General anesthesia by inhalation can occur. Continued exposure may lead to respiratory failure or death. Early symptoms include irritation of nose and throat, vomiting, and irregular respiration, followed by dizziness, drowsiness, and unconsciousness. May cause irritation, redness and pain to the eyes. Irritating to the skin and mucous membranes by drying effect. Can cause dermatitis on prolonged exposure. May be absorbed through skin. **May form explosive peroxides on long standing or after exposure to air or light. This material must be disposed of within six months.**
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Hydroxide	Corrosive	2 mg/m^3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Eye protection that protects against splash, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut.

Disposable gloves that have become contaminated must be removed and discarded, other gloves must be cleaned immediately.

5.5. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride must be conducted in a fume hood with the sash closed as far as the operations will permit. If more than 500 mL of Methylene chloride is spilled, evacuate the area until the area has been cleaned by EH&S.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Canton associate. The situation must be reported **immediately** to a laboratory supervisor and the EH&S Coordinator.

5.8. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus and glass separatory funnels have ground glass joints which can become stuck. Technicians must use Kevlar or other cut/puncture resistant gloves when separating stuck joints.

6. EQUIPMENT AND SUPPLIES

6.1. All glassware is cleaned per SOP NC-QA-014.

6.2. Equipment and supplies for extraction procedures follow.

EQUIPMENT AND SUPPLIES	Sep fun.	Soni	Conc
Separatory Funnel: 2 L	v		
Separatory Funnel Rack	v		
pH indicator paper, wide-range: covers extraction pH	v	v	
Graduated cylinder: 1 liter. (other sizes may be used)	v		
Centrifuge	v	v	
Auto-Shaker	v		
Methylene Chloride Collection Tank	v	v	v
Solvent Dispenser Pump or 100 mL Graduated Cylinder	v	v	v
Beakers: 250 & 400 mL, graduated	v	v	
450mL wide-mouth glass jars	v	v	
1 L amber wide mouth glass jars	v	v	

EQUIPMENT AND SUPPLIES	Sep fun.	Soni	Conc
Balance: >100 g capacity, accurate ?? g		v	
Sonicator (at least 300 watts)		v	
Sonicator horn, 3/4 inch		v	
Kuderna-Danish (K-D) Apparatus: 500 mL			v
Concentrator Tube: 10 mL, attached to K-D with clips			v
Snyder Column: Three-ball macro			v
Water Bath: Heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.			v
Vials: Glass 10 mL capacity with Teflon®-lined screw-cap			v
Nitrogen Blowdown Apparatus			v
Nitrogen: Reagent grade.			v
Culture tubes: 10 mL, 16 mmx100 mm			v
Syringe: 1 mL or positive displacement pipette	v	v	
Glass Wool	v	v	
Funnel: 75 X 75 mm	v	v	v
Disposable Pipettes	v	v	v
Aluminum foil	v	v	v
Paper Towels	v	v	v
Balance: >1400 g capacity, accurate ±.1 g;	v	v	v

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better unless otherwise specified.

REAGENTS	Sep fun.	Soni	Conc
Sodium hydroxide (NaOH), Pellets: Reagent Grade	v		
Sodium hydroxide solution, 10 N: Dissolve 40 g of NaOH in reagent water and dilute to 100 mL.	v		
Sulfuric acid (H_2SO_4), Concentrated: Reagent Grade	v	v	
Sulfuric acid (1:1): Carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.	v	v	
Hydrochloric Acid (HCl)		v	
Organic free reagent water.	v	v	

REAGENTS	Sep fun.	Soni	Conc
Sodium sulfate (Na_2SO_4), Granular, Anhydrous: Purify by heating at 400°C a minimum of two hours.	v	v	v
Extraction/Exchange Solvents: Methylene chloride, hexane, acetonitrile, acetone, toluene, pesticide quality or equivalent	v	v	v
Acetone: Used for cleaning	v	v	v

7.1.1 Potassium hydroxide solution, 37% aqueous solution, (w/v): Dissolve 37 g of potassium hydroxide pellets in reagent water and dilute to 100 mL.
Caution: Considerable heat will be generated. Other volumes of solution may be made up as convenient, in the same proportions.

7.1.2 Sodium sulfate (Na2SO4),anhydrous, granular, acidified: Use approximately 2000g of oven muffled Na2SO4 create a slurry using enough diethyl ether to cover. Add approximately 80 mLs of concentrated H2SO4; mix thoroughly. Place the mixture on steam bath in hood to allow ether to evaporate. Larger or smaller batches may be created using the same reagents in the same proportions. Store in a desiccator. Check the pH of the reagent prior to use by mixing 1 g of the sodium sulfate with 5mL of reagent grade water and testing the pH. The pH must be $\leq$ 4.

7.1.3 Sodium Chloride, NaCl

7.1.4 Diethyl ether, reagent grade

7.1.5 (Trimethylsilyl)diazomethane

7.1.6 Methanol, reagent grade

7.1.7 Silica gel

7.2 Standards

7.2.1 Stock Standards

Stock standards are purchased as certified solutions or prepared from neat standards. Semivolatile stock standards are stored per manufacturer instructions. All stock standards must be protected from light. Stock standard solutions must be replaced after one year (from the time of preparation, if prepared in-house, or from the time the ampoule is opened, if purchased). Standards must be allowed to come to room temperature before use. Additional information can be found in SOP NC-QA-017.

7.2.2. Surrogate Spiking Standards

Prepare or purchase surrogate spiking standards at the concentrations listed in Table 3. Surrogate spiking standards are purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be stored per manufacturer instructions. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.3. Matrix Spiking and Laboratory Control Spiking Standards

The same spiking solution is used for the matrix spike/matrix spike duplicate (MS/MSD) and the Laboratory Control Sample (LCS). Prepare MS/MSD/ LCS spiking standards at the concentrations listed in Tables 4 and 5. Spiking standards are purchased or prepared as dilutions of the stock standards. Spiking solutions must be stored per manufacturer instructions. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.4. Surrogate Standard (see Table 1)

7.2.5. MS/MSD and LCS Standard (see Table 2)

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1 Samples are not chemically preserved.

8.2. Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps and protected from light.

8.3. Holding Times

8.3.1 The holding time for aqueous samples is seven days from sampling to extraction. The holding time for solid and waste samples is 14 days from sampling to extraction.

8.3.2. For TCLP leachates, the holding time is 14 days from sampling to leaching. The holding time for extraction is seven days from when the TCLP leach tumbling has been completed, excluding the filtration step, to extraction. If the filtration step requires extended times, this time counts as part of the seven-day holding time.

8.3.3. The analytical holding time is 40 days from extraction to analysis.

9. QUALITY CONTROL

9.1. Quality Control Batch

9.1.1. The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, a laboratory control sample, and a matrix spike / matrix spike duplicate. If clients specify specific samples for matrix spike/matrix spike duplicate, the batch may contain multiple matrix spike/matrix spike duplicate samples. See Policy QA-003 for further definition of the batch.

9.2. Method Blank (MB)

9.2.1. An MB consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the MB at the same level as the samples. The MB is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data.

9.2.2. Aqueous MBs use 500 mL of reagent water spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.2.3. Solid MBs use approximately 30g of sodium sulfate spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.2.4. TCLP MBs use 100 mL of leachate fluid spiked with the surrogates. The leachate may optionally be diluted to 500 mL with reagent water. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.3. Laboratory Control Sample (LCS)

9.3.1. LCSs are well characterized, laboratory-generated samples used to monitor the laboratory day-to-day performance of routine analytical methods. The LCS spiked with a group of target compounds representative of the method analytes is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure, including any cleanup steps.

9.3.2. The LCS is made up in the same way as the method blank (See Sections 9.2.1 - 9.2.4), but spiked with the LCS standard.

9.4. Surrogates

9.4.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.4.2. Each applicable sample, method blank, laboratory control sample (LCS), and matrix spike/matrix spike duplicate (MS/MSD) is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

9.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.5.1. A matrix spike (MS) is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and MS.

9.6. Control Limits

9.6.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.6.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMs

9.7. Method Detection Limits (MDLs) and MDL Checks

9.7.1. MDLs and MDL Checks are established by the laboratory as described in SOPs CA-Q-S-006 and NC-QA-021.

9.7.2. MDLs are easily accessible via LIMs

9.8. Nonconformance and Corrective Action

9.8.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action.

10. CALIBRATION AND STANDARDIZATION

10.1. On a weekly basis, measure the appropriate volume of solvent into an autovial using a gas-tight syringe that is manufactured to a certified volume delivery tolerance of ±0.01 mL. The "standard" autovial is sealed, and the top and bottom of the meniscus are marked. The autovials containing the sample extracts are then compared against the "standard" vial to ensure the final volume is consistently 1.0 ± 0.01 mL. If a new box of autovials is used, then the steps are repeated to further ensure that variations due to vial size and shape are minimized. A log is kept to track the date of vial preparation.

11. PROCEDURE

Note: Refer to SOP NC-QA-016 for procedures regarding DoD work.

11.1 Preparation of Aqueous Samples

11.1.1 Remove surrogate and matrix spiking solutions from refrigerator and allow to return to room temperature.

11.1.2 Volumetrically transfer 500 mL into a one-liter wide-mouth amber jar using the 500 mL bottling template. For TCLP samples, volumetrically transfer 100mL of sample into the jar, using the 100 mL bottling template and dilute to 500mL with reagent grade water.

11.1.3 Spike each sample, MB, LCS, and MS/MSD with 1.0 mL of DCAA surrogate solution. Spike MS/MSDs and LCS with 1 mL of herbicide matrix spiking solution (refer to Tables 1 and 2).

11.1.4 Add 60-100g of NaCl to samples and QC samples, and shake to dissolve the salt.

11.1.5 Hydrolysis

11.1.5.1 Add approximately 3 mL of 10N NaOH to the sample, stir sample, and check the pH of the sample with pH paper. If the pH of the sample is not $\geq$12, adjust to $\geq$12 by adding more NaOH.

11.1.5.2 Add approximately 100 mL of methylene chloride to the amber jar.

11.1.5.3 Let the sample sit at room temperature for a minimum of 1-2 hours. Shake the contents and vent periodically during this time period.

11.1.5.4 Pour content of amber jar into a pre-rinsed Teflon® sep funnel. Let stand for ten minutes to allow organic layer to completely settle. If an emulsion layer greater than one third of the solvent layer forms, use mechanical techniques to complete the phase separation. Suggested techniques are stirring, filtration through glass wool, and centrifugation.

11.1.5.5 Discard the methylene chloride phase.

11.1.6 Extraction of Acids

11.1.6.1 Add 4-8 mL of 1:1 sulfuric acid to the sample. Seal and shake to mix. Check the pH of the sample with pH paper. If the pH is not $\leq$ 2, add more acid to adjust the pH to $\leq$ 2. ***Caution: Addition of***

acid may cause heat and / or pressure buildup.

11.1.6.2 Add approximately60 mL of diethyl ether to sample, and extract for two minutes by auto rotator, venting as necessary. Allow organic layer to separate from aqueous layer by allowing sample to sit for ten minutes prior to collecting ether layer. Drain aqueous layer into amber liter jar, and collect ether layer in a mason jar containing approximately 20g of acidified anhydrous sodium sulfate.

11.1.6.3 Return the aqueous phase to the separatory funnel, add approximately 60mL diethyl ether, and repeat the extraction procedure a second time, combining the ether extracts. Repeat the extraction a third time with approximately60 mL diethyl ether. Discard the aqueous phase after the third extraction.

11.1.6.4 Allow the extract to remain in contact with the sodium sulfate for at least two hours, shaking periodically (may be left overnight). The drying step is critical. If the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate. The amount of sodium sulfate is sufficient if some free-flowing crystals are visible when the flask or bottle is swirled or shaken.

11.1.6.5 Proceed to Section 11.5, Concentration.

11.2 Extraction of soil and sediment samples

11.2.1 Remove surrogate and matrix spiking solutions from refrigerator, and allow return to room temperature.

11.2.2 Decant and discard any water layer on a sediment/soil sample. Record and document in LIMS if a water layer was discarded. Homogenize the sample by mixing it thoroughly in the container. If this is not possible, place the sample in clean beaker and homogenize. Upon completion of homogenization in beaker, return sample to original container. Discard foreign objects such as sticks, leaves, and rocks, unless extraction of this material is required by client. If the sample consists primarily of foreign materials, consult with the Project Manager.

11.2.3 Weigh 30g (± 0.50g) of moist solid sample into a clean glass jar. Use 30g of sodium sulfate for the MB and LCS. Acidify the sample, MB, LCS, and MS/MSD with approximately5 mL of concentrated HCl.

11.2.4 There must be a small amount of liquid phase. Stir well with a spatula.

11.2.5 Stir with a spatula, and check the pH of the liquid phase. Add more acid if necessary to bring the pH to <2, repeating the stirring after each acid addition.

11.2.6 Dry samples with oven muffled sodium sulfate until the sample is free flowing. The pH of the sodium sulfate must be checked prior to use by taking a few grams and adding to reagent water. The pH should be $\leq$ 7. If not, acidified anhydrous sodium sulfate should be used.

11.2.7 Spike each sample, MB, LCS, and MS/MSD with 1.0 mL of DCAA surrogate solution. Spike MS/MSDs and LCS with 1 mL of herbicide matrix spiking solution (refer to Tables 1 and 2).

11.2.8 Add a minimum of 100 mL of 1:1 methylene chloride/acetone to a clean glass jar.

11.2.9 Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.2.10 Sonicate for three minutes, making sure the entire sample is agitated.

11.2.11 Loosely plug the stem of a 75 mm x 75 mm funnel with glass wool, and/or line the funnel with filter paper. Add 10-20g of anhydrous sodium sulfate to the funnel cup.

11.2.12 Place the prepared funnel on a collection apparatus. If the herbicide esters are to be determined (normally the case), the collection apparatus is glassware suitable for the hydrolysis step--typically a KD flask or Turbovap tube.

11.2.13 Decant and filter extracts through the prepared funnel into the collection apparatus.

11.2.14 Repeat the extraction two more times with additional 100 mL minimum portions of methylene chloride each time. Decant off extraction solvent after each sonication. On the final sonication, pour the entire sample (sediment and solvent) into the funnel and rinse with an additional 10-20 mL of the methylene chloride.

Note: Alternatively, the three extracts may be collected together and then filtered through the sodium sulfate.

11.3 Hydrolysis

11.3.1 Add 5 mL of 37% aqueous potassium hydroxide and 30 mL of reagent grade water to the extract. Shake the sample vigorously for 30 seconds, and let stand for ten minutes. Check the pH with pH paper. If the pH is not $\geq$12, adjust with additional KOH.

11.3.2 Heat on a water bath at 94 ± 4°C until the organic layer is completely evaporated and the Snyder column has stopped chattering.

11.3.3 Before transferring the extract to a separatory funnel, assure pH is still ≥12. If not, adjust by adding more 37%KOH and 50mL of methylene chloride, and return to steam bath for 15 minutes. Verify the pH is ≥12 when finished.

11.3.4 Transfer the solution to a separatory funnel and extract serially three times with 60 mL portions of methylene chloride. **Discard the methylene chloride phase.** After the third time, let stand for ten minutes prior to discarding the last layer of methylene chloride to ensure all methylene chloride is removed from the aqueous layer. The aqueous layer will contain the herbicides as long as the aqueous layer remains basic. If an emulsion layer greater than one-third of the solvent layer forms, use mechanical techniques to complete the phase separation. Suggested techniques are stirring, filtration through glass wool, and centrifugation

11.4 Extraction of Acids

11.4.1 Add 4-8 mL of 1:1 sulfuric acid to the sample. Seal and shake to mix. Check the pH of the sample with pH paper. If the pH is not ≤2, add more acid to adjust the pH to ≤ 2. ***Caution: Addition of acid may cause heat and / or pressure buildup.***

11.4.2 Add 60 mL of diethyl ether to sample and extract serially three times for two minutes by auto rotator, venting as necessary. Allow organic layer to separate from aqueous layer by allowing sample to sit for ten minutes prior to collecting ether layer. Drain aqueous layer into a jar, and collect ether layer in a mason jar containing approximately 20g of acidified anhydrous sodium sulfate.

11.4.3 Return the aqueous phase to the separatory funnel, add 60 mL diethyl ether, and repeat the extraction procedure a second time, combining the ether extracts. Repeat the extraction a third time with 60 mL diethyl ether. Discard the aqueous phase after the third extraction.

11.4.4 Allow the extract to remain in contact with the sodium sulfate for at least two hours (may be left overnight). The drying step is critical. If the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate. The amount of sodium sulfate is sufficient if some free-flowing crystals are visible when the flask or bottle is swirled or shaken.

11.4.5 Proceed to Section 11.5, Concentration.

11.5 Concentration

11.5.1 Transfer the ether extract into a 500 mL K-D flask **equipped with a 10 mL concentrator tube.** Crush the caked sodium sulfate during transfer. Rinse the flask or bottle with 20-30 mL ether to complete transfer.

11.5.2 Attach a three-ball Snyder column to the K-D apparatus, pre-wet the column with a few mL of ether from the top, and place the apparatus on a water bath at approximately 65°C. At the proper rate of distillation, the balls of the column will chatter, but the chambers will not flood. When the apparent volume reaches approximately 20 mL, exchange with approximately 18 mL of Hexane, remove from the water bath, and allow to completely cool.

11.5.3 Carefully disassemble the concentrator tube, and rinse the lower glass joint with a small amount of diethyl ether.

11.5.4 Place the extracts on the nitrogen blowdown and allow to concentrate to 2 mL. **Note:** For Non-TCLP extracts, the extracts must be quantitatively transferred to a test tube prior to being placed on the blowdown.

11.5.5 The extract is now ready for esterification by (Trimethylsilyl)diazomethane (Section 11.7).

11.6 Esterification by (Trimethylsilyl)diazomethane

11.6.1 Add approximately 200 uL of methanol to the extract followed by approximately 200 uL of (Trimethylsilyl)diazomethane solution. The extract will turn a yellow color. If this does not occur, add an additional approximate 200 uL aliquots of (Trimethylsilyl)diazomethane solution until the yellow color persists. Check the sample every 15 minutes for yellow color. If the yellow disappears in this time frame, add an additional approximate 200 uL aliquots of (Trimethylsilyl)diazomethane solution until the yellow color persists.

11.6.2 Allow the extract to stand for at least one hour at room temperature to allow the methylation reaction to occur. The reaction is halted with the addition of silica gel. The extract is then brought up to a final volume of 10 mL with hexane by visually comparing it to a calibrated collection tube. The sample is now ready for analysis.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

13.1.1. Each laboratory must make a one-time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests, it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.1.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples including sample preparation. The concentration of the QC check sample must be equivalent to a mid-level calibration.

13.1.3. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs.

13.1.4. If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.2. Training Qualification

13.2.1. The Group/Team Leader has the responsibility to ensure an analyst who has been properly trained in its use and has the required experience performs this procedure.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files

14. POLLUTION PREVENTION

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method the policies in Section 13 of the Corporate Environmental

Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2. The following waste streams are produced when this method is carried out.

15.2.1. **Aqueous acidic waste.** These wastes are disposed of in the liquid-liquid separation unit.

15.2.2. **Non-hazardous sodium sulfate**. Non-hazardous substances can be disposed of in the regular trash.

16. REFERENCES

16.1. References

16.1.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Chlorinated Herbicides, Method 8151A

16.1.2. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.3. TestAmerica A Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.4. Corporate Quality Management Plan (CQMP), **current version**

16.1.5. Revision History

Historical File:		Revision 0: 10/17/06		Revision 4: 04/20/10
		Revision 1: 09/11/07		Revision 5-A: 05/29/12
		Revision 2: 08/20/08		
		Revision 3: 01/07/09		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-016

16.2.6. Gas Chromatographic Analysis based on Methods 8015B and 8015C, NC-GC-043

16.2.8 Standards and Reagents, NC-QA-017

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Section 7.5 of Method 8151A lists diazomethane and PFB for the esterification process. The laboratory is using (Trimethylsilyl)diazomethane and Boron Trifluoride for the esterification process.

17.2. Tables

TABLE 1
Herbicide Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume (mL)
Herbicides	Herbicides TCLP	1.0
Herbicides	Herbicides Soil & Water	1.0

TABLE 2
Herbicide Matrix Spike/Matrix Spike Duplicate (MS/MSD) and Laboratory Control Sample (LCS) Solutions

Analyte Group	Matrix Spike Solution ID	Volume (mL)
Herbicides	Herbicides EA	1.0
Herbicides	Herbicides TCLP	1.0

TABLE 3
Herbicide Surrogate Spike Components

Type	Compounds	Solvent	Conc (ug/mL)
Herbicides TCLP	2,4-DCAA	Acetone	2
Herbicides	2,4-DCAA	Methanol	20

[1]The surrogate is spiked as the free acid.

TABLE 4
Herbicide Matrix Spike/Matrix Spike Duplicate (MS/MSD) Components

Type	Compounds[1]	Compounds[1] Solvent	EA Conc. (ug/mL)
Herbicides MS	2,4-D	Methanol	20
	2,4-DB		20
	2,4,5-TP (Silvex)		5
	Dalapon		10
	Dicamba		10
	Dichloroprop		20
	Dinoseb		3
	2,4,5-T		5
	MCPA		2000
	MCPP		2000
	Pentachlorophenol		2.5

[1]The herbicide spiking solution contains the herbicides as the free acids.

TABLE 5

Herbicide TCLP Matrix Spike/Matrix Spike Duplicate (MS/MSD) Components

Type	Compounds	Compounds Solvent	TCLP Conc. (ug/mL)
Herbicides TCLP	2,4-D	Acetone	2
	2,4,5-T		0.5

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Canton

SOP No. NC-IP-010, Rev. 4
Effective Date: 9/4/13

Title: ACID DIGESTION FOR SOLID SAMPLES

[Method: SW846 Method 3050B]

Approvals (Signature/Date):

Technology Specialist	09/04/13 Date	Health & Safety Coordinator	09/04/13 Date
Quality Assurance Manager	08/28/13 Date	Laboratory Director	09/04/13 Date

This SOP was previously identified as SOP No. NC-IP-010, Rev 2, dated 08/12/10

Facility Distribution No. _____ **Distributed To: _____**

TABLE OF CONTENTS

LIST OF APPENDICES:

1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of soil samples for the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) and Inductively Coupled Plasma-Mass Spectrometry (ICPMS) as specified in SW846 Method 3050B.

1.2. Samples prepared by the protocols detailed in this SOP may be analyzed by ICP or ICPMS for the elements listed in Table I (Appendix A). Other elements and matrices may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.

1.3. This method is not a total digestion, but will dissolve almost all metals that could become "environmentally available". By design, metals bound in silicate structures are not dissolved by this procedure, as they are not usually mobile in the environment. This SOP can be applied to metals in solids, sludges, wastes, sediments, biological samples, and wipes.

1.4. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. A representative 1 gram (wet weight) portion of sample is digested in nitric acid and hydrogen peroxide. The digestate is refluxed with hydrochloric acid for ICP and ICPMS analysis. The digestates are then filtered and diluted to 100 mL for subsequent analysis.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), latest version.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include metallic or metal-containing lab-ware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination, and take appropriate measures to minimize or avoid them. All glassware is cleaned per SOP NC-QA-014.

4.2. The entire work area, including the bench top and fume hood, must be thoroughly

cleaned on a routine schedule in order to minimize the potential for environmental contamination.

4.3. Boron from the glassware may leach into the sample solution during, and following, sample processing. For critical low-level determinations of boron, only quartz and/or plastic lab-ware are recommended.

4.4. Visual interferences or anomalies, such as foaming, emulsions, precipitates, etc., must be documented.

4.5. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be re-prepared. Antimony is easily lost by volatilization from hydrochloric media.

4.6. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.5 The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples must be done in a fume hood. The analyst must also be aware of the potential for a vigorous reaction.

5.6 Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.7 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8 Always carry bulk concentrated acid bottles in appropriate impact proof containers.

5.9 Acid/peroxide spills must be neutralized immediately, flushed with water and cleaned up using appropriate spill kits.

5.10 Discard chipped or broken beakers to prevent injury. Chipped glassware may be fire

polished as an alternative to disposal.

6. EQUIPMENT AND SUPPLIES

6.1. Hot plate, digestion block, steam bath, or other heating source capable of maintaining a temperature of 91-99°C

6.2. Calibrated thermometer that covers a temperature range of 0-110°C

6.3. Vapor recovery device (Watch glasses, ribbed or other device)

6.4. Whatman No. 41 filter paper or equivalent

6.5. Funnels or equivalent filtration apparatus

6.6. Analytical balance capable of accurately weighing to the nearest 0.01 grams

6.7. Repeaters or suitable reagent dispensers

6.8. Calibrated automatic pipettes with corresponding pipette tips: 100uL, 500uL, 1mL-5mL

6.9. Class A volumetric flasks

6.10. Plastic digestate storage bottles, such as Corning Snap Seals™ (may be used if their accuracy is documented and is better than 2%)

6.11. Boiling Stones: Ultra Pure PTFE or equivalent

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks (MB) as defined in the determinative SOPs.

7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Additional information can be found in SOP NC-QA-017.

7.3. Working ICP LCS/ MS solution: Prepare the ICP LCS/ MS working spike solutions from custom stock standards to the final concentration listed in Table II. The working spike must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working ICP LCS/MS solution must be made fresh every six months. Refer to the reagent module in LIMS for details on standard preparation.

7.4. ICPMS LCS/MS solution: LCS/MS solutions are custom made so the final concentrations after spiking equals the concentrations listed in Table III.

7.4.1. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom solution, the individual facility must purchase a solution from the designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.5. Nitric acid (HNO_3), concentrated, trace metal grade or better

7.6. Nitric acid, 1:1 - dilute concentrated HNO_3 with an equal volume of reagent water

Note: When preparing diluted acids, always add acid to water. If the water is added to the acid a violent reaction may occur.

7.7. Hydrochloric acid (HCl), concentrated, trace metal grade or better

7.8. 30% Hydrogen peroxide (H_2O_2), Ultrapure grade

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Solid samples are collected and stored in wide-mouth glass jars with PTFE-lined lids. A minimum of 10 g must be collected.

8.2. Sample holding time for metals included under the scope of this SOP is 180 days from the date of sample collection to the date of analysis.

8.3. Soil samples do not require preservation.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using Method 3050B the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The result of the MDL

determination must be below the TestAmerica Canton reporting limit. Criteria for DoD work is noted in SOP NC-QA-016.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which must contain all the analytes of interest. The results of the initial demonstration study must be acceptable before analysis of samples may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.2. Preparation Batch

9.2.1. A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain an MB, an LCS, and an MS/MSD. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count

9.3.1. Laboratory generated QC samples (method blanks LCS MS/MSD) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

9.4. Method Blank (MB)

9.4.1. One MB must be processed with each preparation batch. The MB consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Criteria for the acceptance of MB are contained within the individual analytical method SOPs. If the MB does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be re-digested.

9.4.2. The MB is prepared by weighing a 1g aliquot of boiling chips. The MB is then processed as described in Section 11.9.

9.5. Laboratory Control Sample (LCS)

9.5.1. One LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOPs. Corrective action when LCS results fail to meet control limits must be repreparation and reanalysis of the batch. Table II provides the details regarding the stock, working standards, and final spike concentrations for ICP and ICPMS. Refer to Section 7 for instructions on preparation of the LCS.

9.5.2. The ICP LCS is prepared by spiking a 1g aliquot of boiling chips with 2 mL of the working LCS/ MS spike solution (Section 7.4). The ICPMS LCS is prepared by spiking a 1g aliquot of boiling chips with 1 mL of the LCS/MS solution (Section 7.4). The LCS is then processed as described in Section 11.9.

9.6. Additional information on QC samples can be found in QA Policy QA-003. Ohio VAP projects must reference this SOP instead of policy QA-003 for information on QC samples.

9.7. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.7.1. One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. An MSD is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis. If any analyte recovery or RPD falls outside the control limits, the recovery of that analyte must be in control for the LCS. If the recovery of the LCS is outside of control limits, corrective action must be taken. Corrective action must include repreparation and reanalysis of the batch. Corrective action when the MS results fail to meet control limits does not include repreparation of samples unless the results indicate that a spiking error may have occurred. Client specific samples may require corrective action. Such action is noted in the project narrative. Tables II and III provides the details regarding the stock, working standards and final matrix spike concentrations for ICP and ICPMS. Refer to Section 7 for instructions on preparation of the working matrix spike solutions.

9.7.2. The ICP MS/MSD is prepared by spiking a 1g aliquot of sample with 2 mL of the working LCS/MS spike solution (Section 7). The ICPMS MS/MSD is prepared by spiking a 1g aliquot of sample with 1 mL of the LCS/MS solution (Section 7.4). The MS/MSD is then processed as described in Section 11.9.

10. CALIBRATION AND STANDARDIZATION

10.1. Hot block temperature must be verified daily for each unit used, and must be recorded in a hot block temperature log.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo (NCM).

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described. Deviations are not allowed for Ohio VAP projects.

11.3. The heating procedures are carried out in a properly functioning hood.

11.4. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample. LIMS provides sample labels to reduce transcription errors.

11.5. Samples are typically logged in as soils. Wastes, such as organic liquids or sludges and tissues (animal/vegetable), are usually logged in as solids. When initiating prep, examine the sample to see if the sample matches the matrix designation.

11.6. If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab.

11.7. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards. Refer to Appendix B for details.

11.8. Preparation of Soils, Sediments, and Sludges for Analysis by ICP and ICPMS

11.8.1. Mix sample thoroughly by stirring with a clean plastic or wooden spoon or spatula.

11.8.2. For each digestion procedure required (i.e., ICP or ICPMS), weigh a 1g portion of solid and record the exact weight to the nearest 0.01g. A 2g sample size may also be used if needed to meet the reporting limits.

Note: Wipe samples are not weighed. The entire wipe is used.

11.8.3. Measure additional aliquots of the designated sample(s) for the MS/ MSD analyses. MS/MSD samples must be weighed to the exact nominal weight due to a limitation of the LIMS system.

11.8.4. Add 10 mL of 1:1 HNO_3 and mix the sample.

11.8.5. Heat sample to 95° ±4° C and reflux for 10 minutes without boiling, using a vapor recovery device.

Note: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY during any part of the digestion. Doing so will result in the loss of analyte and the sample must be re-prepared.

11.8.6 Add 5 mL of concentrated HNO_3.

11.8.7 Reflux at 95° ±4° C for 30 minutes. (Add reagent water, as needed, to ensure that the volume of solution is not reduced to less than 5 mL.)

11.8.8 Add approximately 2 mL of reagent water and 1 mL of 30 % H_2O_2. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

11.8.9 Continue adding 30% H_2O_2 in 1 mL aliquots until effervescence is minimal or sample appearance is unchanged. Make sure effervescence subsides before each addition of H_2O_2.

Note: Do not add more than a total of 10 mL of 30 % H_2O_2.

11.8.10 Continue heating at 95° ± 4° C until the volume is reduced by cooking two hours or to approximately 5-10 mL.

11.8.11 Add 10 mL of concentrated HCL and reflux for an additional 15 minutes without boiling.

11.8.12 Allow the sample to cool.

11.8.13 Filter sample through Whatman 41 filter paper or equivalent, that has been rinsed with deionized water, into a measuring bottle (for example, Corning Snap Seals™). These may be used if their accuracy is documented and is better than ± 2%. Rinse sample container and filter paper with reagent water to ensure complete sample transfer.

11.8.14 Dilute sample to 100 mL with reagent water into a 120 mL graduated Snap Seal. The sample is now ready for analysis.

11.9. Analytical Documentation

11.9.1 Record all analytical information in LIMS including the analytical data from standards, blanks, LCSs, and MS/MSDs, Any corrective actions or modifications to the method must be noted in an NCM

11.9.2 All standards and reagents are logged into the LIMS standards and reagents module. All standards are assigned a unique number for identification.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. Acceptance criteria are given in the determinative SOPs.

13.2. The initial demonstration study as detailed in Section 9.1.2 must be acceptable before the analysis of field samples under this SOP may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

13.3. Training Qualification

The Group/Team Leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste must be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2. Waste Streams Produced by the Method

15.2.1. The following waste streams are produced when this method is carried out.

15.2.1.1. Acidic waste containing nitric acid generated by the extraction. This

waste is disposed of in a designated container labeled "Acid Waste".

15.2.1.2. Contaminated disposable materials utilized for the analysis. This waste is disposed of in a designated container labeled "Solid Waste".

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996, Method 3050B

16.1.2. TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.4. Corporate Quality Management Plan (CQMP), current version

16.1.5. Revision History

Historical File:		Revision 2.1: 02/11/00		Revision 0: 07/18/08 (NC-IP-010)
(formerly CORP-IP0002NC)		Revision 2.2: 09/25/01		Revision 1: 01/07/09
		Revision 2.3: 01/18/02		Revision 2: 08/12/10
		Revision 2.4: 02/19/03		
		Revision 2.5: 12/02/04		
		Revision 2.6: 07/29/07		

16.2. Associated SOPs and Policies, current version

16.2.1. Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Methods 6010B and 200.7,
NC-MT-012

16.2.2. Inductively Coupled Plasma-Mass Spectrometry, EPA Methods 6020 and 200.8,
NC-MT-002

16.2.3. TestAmerica North Canton Quality Control Program, QA-003

16.2.4. Glassware Washing, NC-QA-014

16.2.5. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.6. Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.7. Supplemental Practices for DOD Project Work, NC-QA-016

16.2.8. Standards and Reagents, NC-QA-017

16.2.9. Subsampling, NC-IP-001

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Method Deviations

17.1.1. The laboratory uses the same preparation procedure for Method 6010B and 6020. Hydrochloric acid can be used for Method 6020 due to the collision cell technology on newer instruments. Due to the potential chloride interferences, and possible inability to analyze for arsenic and tin, the laboratory must follow the instrument manufacturer guidelines pertaining to the use of HCL and ICP/MS analyses.

APPENDIX A: TABLES

TABLE I

Method 3050B Approved Analyte List

Element	Symbol	CAS Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Boron	B	7440-42-8
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Lithium	Li	7439-93-2
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Tin	Sn	7440-31-5
Titanium	Ti	7440-32-6
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

TABLE II

ICP Soil Matrix Spike (MS) and Laboratory Control Sample (LCS) Levels

Element	Working LCS/MS Standard (mg/L)	Soil MS/LCS Level * (mg/kg)
Aluminum	100	200
Antimony	25	50
Arsenic	100	200
Barium	100	200
Beryllium	2.5	5
Cadmium	2.5	5
Calcium	2500	5000
Chromium	10	20
Cobalt	25	50
Copper	12.5	25
Iron	50	100
Lead	25	50
Magnesium	2500	5000
Manganese	25	50
Molybdenum	50	100
Nickel	25	50
Potassium	2500	5000
Selenium	100	200
Silver	2.5	5
Sodium	2500	5000
Thallium	100	200
Vanadium	25	50
Zinc	25	50
Boron	50	100
Tin	100	200
Titanium	50	100
Silicon	50	100
Silica	107	214

* Final soil spike concentration based on the addition of 2.0 mL working spike (Section 7.3) to 1.0 g of sample/100 mL final volume (assumes 100% solids).

TABLE III

ICPMS Soil Matrix Spike (MS) and Laboratory Control Sample (LCS) Levels

Element	Working LCS/MS Standard (mg/kg)	Soil MS/LCS Level * (mg/kg)
Aluminum	1000	1000
Antimony	10	10
Arsenic	10	10
Barium	10	10
Beryllium	10	10
Cadmium	10	10
Calcium	1000	1000
Chromium	10	10
Cobalt	10	10
Copper	10	10
Iron	1000	1000
Lead	10	10
Lithium	10	10
Magnesium	1000	1000
Manganese	10	10
Molybdenum	10	10
Nickel	10	10
Potassium	1000	1000
Selenium	10	10
Silver	10	10
Sodium	1000	1000
Strontium	10	10
Thallium	10	10
Vanadium	10	10
Zinc	10	10
Boron	10	10
Tin	10	10
Titanium	10	10

* Final soil spike concentration based on the addition of 1.0 mL working spike (Section 7.4) to 1.0 g of sample/100 mL final volume (assumes 100% solids).

APPENDIX B. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware must be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the Metals Lab. All work areas must be kept scrupulously clean.

Powdered gloves must not be used in the Metals Lab since the powder contains zinc, as well as other metallic analytes.

Glassware must be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Canton

SOP No. NC-IP-011, Rev. 4
Effective Date: 06/28/13

Title: ACID DIGESTION FOR AQUEOUS SAMPLES

[Methods: SW846 3005A, 3010A, and MCAWW 200 Series]

Approvals (Signature/Date):

Susan M. Girard	06/25/13		06/25/13
Technology Specialist	Date	Health & Safety Coordinator	Date
Rebecca Strait	06/25/13	[redacted]	06/27/13
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. NC-IP-011, Rev 3-A, dated 06/28/12

Facility Distribution No. _____ **Distributed To: _____**

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LIST OF APPENDICES

1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of aqueous samples for the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP), and Inductively Coupled Plasma-Mass Spectrometry (ICPMS) using the MCAWW 200 series methods (NPDES) and SW846 Methods 3005A, and 3010A.

1.2. The applicability of each of these preparation protocols to specific analytes is detailed in Tables I and II (Appendix A). Additional elements may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.

1.3. This SOP provides procedures applicable to the preparation of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, certain aqueous sludges, and leachates/extracts.

1.4. SW846 Method 3005A / MCAWW Method 200.8 are used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by ICP and ICPMS.

1.5. MCAWW Method 200.7 is used to prepare surface water, domestic and industrial waste samples for total, total recoverable, and dissolved metals determination by ICP.

1.6. SW846 Method 3010A is used to prepare aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for total metals analysis by ICP.

2. SUMMARY OF METHOD

2.1. Method 3005A / Method 200.7 / Method 200.8 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP and ICPMS

2.1.1 A representative aliquot of sample is heated with nitric and hydrochloric acids and reduced to a low volume. The digestate is filtered (if necessary) and diluted to volume.

2.2. Method 3010A / Method 200.7 / - Preparation for Total Metals Analysis by ICP

2.2.1 A representative aliquot of sample is refluxed with nitric acid. After the digestate has been reduced to a low volume, it is refluxed with 1:1 hydrochloric acid, filtered (if necessary), and diluted to volume.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include metallic or metal-containing lab ware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. All glassware is cleaned per SOP NC-QA-014.

4.2. The entire work area, including the bench top and fume hood, must be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix B for additional contamination control guidelines.

4.3. Boron from the glassware will migrate into the sample solution during and following sample processing. For critical low-level determinations of boron, it is recommended quartz or plastic lab ware be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrices may not be digested using these methods if they are not soluble with acids.

4.5. Visual interferences or anomalies (such as dilution due to oily matrix) must be documented.

4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be re-prepared. Antimony is easily lost by volatilization from hydrochloric acid media.

4.7. Precipitation of silver chloride (AgCl) may occur when chloride ions and high concentrations of silver (i.e., greater than 1 mg/L) are present in the sample.

4.8. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Samples that contain high concentrations of carbonates, or organic material or samples that are at elevated pH, can react violently when acids are added.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.5. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples must be done in a fume hood. The analyst should also be aware of the potential for a vigorous reaction.

5.6. Exposure to chemicals must be maintained **as low as reasonably achievable.** All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8. Always carry bulk concentrated acid bottles in appropriate impact proof containers.

5.9. Acid spills must be neutralized immediately, flushed with water, and cleaned up using appropriate spill kits.

5.10. Discard chipped or broken beakers to prevent injury. Chipped glassware may be fire-polished as an alternative to disposal.

6. EQUIPMENT AND SUPPLIES

6.1. Hot plate, digestion block, or other adjustable heating source capable of maintaining a temperature of 90-95°C

6.2. Calibrated thermometer that covers a temperature range of 0-110°C

6.3. Griffin beakers of assorted sizes or equivalent: Equivalent containers may be disposable digestion cups for digestion blocks, which are certified by the manufacturer and calibrated by the laboratory.

6.4. Watch glasses, ribbed or equivalent

6.5. Whatman No. 41 filter paper or equivalent

6.6. Funnels or equivalent filtration apparatus

6.7. Graduated cylinder or equivalent capable of measuring 50 mL within 3% accuracy

6.8. Analytical balance capable of accurately weighing to the nearest 0.01 grams

6.9. Repeaters or suitable reagent dispensers

6.10. Calibrated automatic pipettes with corresponding pipette tips

6.11. Class A volumetric flasks

6.12. pH indicator strips (pH range 0 - 6)

6.13. Plastic digestate storage bottles, such as Corning Snap Seals (may be used if their accuracy is documented and is better than 2%)

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks (MB) as defined in the determinative SOPs.

7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Additional information can be found in SOP NC-QA-017.

7.3. Working ICP laboratory control sample (LCS)/matrix spike (MS) spike solution: Prepare the ICP LCS/MS working spike solution from custom stock standards to the final concentration listed in Table III. The working spike must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working ICP LCS solution must be made fresh every six months.

7.4. The ICPMS LCS/MS solution is provided directly by the vendor. No further standard preparation is necessary.

7.5. The TCLP MS working spike solution is provided directly by the vendor, no further standard preparation is necessary. Refer to Table V for final digestate spike concentrations.

7.6. The LCS/MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom solution, the individual facility must purchase a solution from the designated

vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.7. Aqueous LCS and MS samples are prepared as described in Sections 9.4 and 9.6. Refer to Tables III and IV (Appendix A) for details regarding the stock, working standard and final digestate spike concentrations for ICP and ICPMS LCS and MS preparations.

7.8. Nitric acid (HNO_3), concentrated, trace metal grade or better

7.9. Hydrochloric acid (HCl), concentrated, trace metal grade or better

7.10. Hydrochloric acid, 1:1 - dilute concentrated HCl with an equal volume of reagent water.

Note: When preparing diluted acids, always add acid to water. If the water is added to the acid, a violent reaction may occur.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and must be stored in either plastic or glass. If boron is to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. Metals samples that are preserved at the laboratory must be held for 24 hours before digestion. For metals samples that require preservation, the Sample Receiving Department must note the time of acid addition.

Note: If the samples are preserved the same day of collection, the 24-hour waiting period is not required

8.4. For dissolved metals analysis, the samples must be filtered through a 0.45 um filter prior to preservation. Filtration must be done in the field. In the event that samples are not field filtered, filtration occurs in the laboratory prior to preparation.

9. QUALITY CONTROL

9.1. Preparation Batch: A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank (MB), an LCS, and a matrix spike/matrix spike duplicate (MS/MSD). In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate (DU) in place of the MS/MSD If clients specify specific samples for MS/MSDs, the batch may contain multiple MS/MSD pairs.

9.2. Sample Count: Laboratory generated QC samples (MB, LCS, MS/MSD) are not included in the sample count for determining the size of a preparation batch.

9.3. Method Blank (MB): One MB must be processed with each preparation batch. The MB consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

9.3.1 Aqueous and TCLP MBs are prepared by taking 50 mL of reagent water through the appropriate procedure as described in Section 11.

9.3.2 TCLP Leachate Blanks (LBs) are prepared by taking 50 mL of leachate fluid through the appropriate procedure as described in Section 11.

9.4. Laboratory Control Sample (LCS): One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOPs. Refer to Sections 7.3 and 7.4 for instructions on preparation of the aqueous LCS spike solution.

9.4.1 The aqueous LCS is prepared by spiking a 50 mL aliquot of reagent water with 1.0 mL for ICP and 0.5 mL for ICPMS of the working LCS/MS spike solution

(Sections 7.3 or 7.4). The LCS is then processed through the appropriate procedure as described in Section 11.

Note: TCLP LCS is prepared by spiking 50 mL of leachate fluid with 1.0 mL for ICP, and 0.5 mL for ICPMS of the working LCS/MS solution and taking it through the appropriate procedure as described in Section 11.

9.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD): One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the matrix spike) prepared and analyzed along with the sample and MS. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.5.1 The aqueous MS sample is prepared by spiking a 50 mL aliquot of a sample with 1.0 mL for ICP and 0.5 mL for ICPMS of the working LCS/MS spike solution (Sections 7.3 or 7.4). The MS sample is then processed as described in Section 11.

9.5.2 The TCLP MS/MSD sample is prepared by spiking a 50 mL aliquot of a leachate with 0.5 mL of the working TCLP spike solution (Section 7.5). The MS/MSD sample is then processed as described in Section 11.

Note: The TCLP matrix spike standard must be added prior to preservation of the leachate.

Note: If analytes outside of the RCRA list are requested, 1 mL of additional spiking solution(s) is added.

9.6 Additional information on QC samples can be found in QA Policy QA-003.

9.7 Control Limits

9.7.1 Control limits are established by the laboratory as described in SOP NC-QA-018.

9.7.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMS.

9.8 Method Detection Limits (MDLs) and MDL Checks

9.8.1 MDLs and MDL Checks are established by the laboratory as described in SOPs NC-QA-021 and CA-Q-S-006.

9.8.2 MDLs are easily accessible via LIMS.

9.9 Nonconformance and Corrective Action

9.9.1 Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action. Procedural deviations are not allowed for Ohio VAP projects.

10. CALIBRATION AND STANDARDIZATION

10.1 The hot plate/hot block temperature must be verified daily for each hotplate used, and must be recorded on a hotplate/hot block temperature log.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure must be completely documented using a Nonconformance Memo. The NonconformanceMemo must be filed in the project file. Procedural deviations are not allowed for Ohio VAP projects.

11.2. All digestion procedures must be carried out in a properly functioning hood.

11.3. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample.

11.4. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable)are usually logged in with solid test codes. When initiating prep, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge like, organic liquid, lots of sediment, etc.), contact the lab supervisor or project manager for further instructions. In some cases, it may be more appropriate to process these samples as solids.

11.5. If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab.

11.6. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

11.7. For DoD work, refer to SOP NC-QA-016 for specific details.

11.8. The following procedure must be followed for all aqueous sample preparations.

11.8.1 Mix sample by shaking the container.

11.8.2 Measure 50 mL of the sample into a calibrated digestion tube. (Beakers may be used for oil matrices.)

Note: For samples with particulate matter, the aliquot may be taken through a repeated series of shake and pour steps.

11.8.3 Measure two extra aliquots of sample selected for the MS/MSD analysis. Spike each aliquot with the appropriate spiking solutions (Sections 7.3 to 7.5 and 9.6).

11.8.4 Measure 50 mL of reagent water into a calibrated digestion tube for the MB .

11.8.5 Measure 50 mL of reagent water into a calibrated digestion tube for the LCS and add the appropriate spiking solutions (Sections 7.3 to 7.5 and 9.6).

11.9. Method 3005A / Method 200.7 / Method 200.8 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP / ICPMS

11.9.1 To the sample container, add 1 mL of concentrated HNO_3 and 2.5 mL of concentrated HCl.

11.9.2 Cover with ribbed watch glass.

11.9.3 Heat at 90-95°C until volume is reduced to between 15 and 20 mL. For DoD samples, the final volume is reduced to between 5 and 10 mL.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

11.9.4 Cool the beaker in a fume hood.

11.9.5 Filter sample, if insoluble materials are present, through Whatman 41 filter paper into a plastic storage container, such as a Corning Snap Seal™

Note: If any samples in a preparation batch are filtered, the MB and LCS associated with that batch must also be filtered.

11.9.6 Rinse container and filter paper with reagent water to ensure complete sample transfer.

11.9.7 Adjust the final volume to 50 mL with reagent water in the Snap Seal™ container if the digestate was filtered or in the hot block digestion tube if filtering was not necessary. The sample is now ready for analysis.

11.10. Method 3010A / Method 200.7 - Preparation for Total Metals Analysis by ICP Spectroscopy

11.10.1. To the sample container, add 3.0 mL of concentrated HNO_3.

11.10.2. Cover with ribbed watch glass.

11.10.3. Place container on hot block 90-95°C, and evaporate to a volume of 15-20 mL, while ensuring that no portion of the bottom of the beaker is allowed to go dry. For DoD projects, the sample is evaporated to 5-10 mL.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

11.10.4. Add 5 mL of 1:1 HCl.

11.10.5. Cover and reflux for an additional 15 minutes to dissolve precipitate or residue. Cool in a fume hood.

11.10.6. Filter sample, if insoluble materials are present, through Whatman 41 filter paper into a plastic storage container, such as a Corning Snap Seal™.

Note: If any samples in the QC batch are filtered, the MB and LCS associated with that batch must also be filtered.

11.10.7. Rinse container and filter paper with reagent water to ensure complete sample transfer.

11.10.8. Adjust final volume to 50 mL with reagent water in the Snap Seal™ container if the digestate was filtered, or in the hot block digestion tube if filtering was not necessary. The sample is now ready for analysis.

11.11. Analytical Documentation

11.11.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.11.2. Record all standards and reagents in the LIMS Reagents module. All standards and reagents are assigned a unique number for identification

11.11.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.11.4. Record all sample results and associated QC into LIMS. Level I and Level II review is performed in LIMS.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Initial Demonstration

13.1.1. Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC Check samples

13.1.2. Four aliquots of the QC check sample are analyzed using the same

procedures used to analyze samples, including sample preparation.

13.1.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs.

13.2. Training Qualification

13.2.1 The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2 Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste must be disposed of in accordance with Federal, State and Local laws and regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees must abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2. Waste Streams Produced by the Method

15.2.1 The following waste streams are produced when this method is carried out.

15.2.1.1 Acidic waste containing nitric acid generated by the extraction: This waste is disposed of in the designated container labeled "Acid Waste".

15.2.1.2 Contaminated disposable materials utilized for the analysis. This waste is disposed of in a designated container identified as "Solid Waste".

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks followed by annual refresher training.

16. REFERENCES

16.1. References

16.1.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, Revision 1, July 1992. Methods 3005A and 3010A

16.1.2 Methods for the Chemical Analysis of Water and Waste (MCAWW), 1983

16.1.3 TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.4 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.5 Corporate Quality Management Plan (CQMP), current version

16.1.6 Revision History

Historical File:		Revision 1.2: 03/20/00		Revision 0: 01/07/09 (NC-IP-011)
(formerly CORP-IP-0003NC)		Revision 1.3: 09/25/01		Revision 1: 01/28/10 (NC-IP-011)
		Revision 1.4: 02/19/03		Revision 2: 05/17/11
		Revision 1.5: 12/07/04		Revision 3-A: 06/28/12
		Revision 1.6: 02/07/07		

16.2. Associated SOPs and Policies, current version

16.2.1 TestAmerica QC Program, QA-003

16.2.2 Glassware Washing, NC-QA-014

16.2.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4 Method Detection Limits and Instrument Detection Limits, NC-QA-021 and CA-Q-S-006

16.2.5 Supplemental Practices for DoD Project Work, NC-QA-016

16.2.6 Standards and Reagents, NC-QA-017

16.2.7 Subsampling, NC-IP-001

16.2.8 Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analyses, SW846 Methods 6010B, 6010C, and 200.7, NC-MT-012

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference methods

17.1.1 Modifications applicable to SW-846 reference methods

17.1.1.1 The referenced methods as well as Table 3-1 of SW-846 refer to the use of a 100 mL aliquot for digestion. This SOP requires the use of a 50 mL sample size to reduce waste generation. The use of reduced sample volumes are supported in EPA's document "Response to Public Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition" dated November 3, 1994. This document stated "flexibility to alter digestion volumes is addressed and "allowed" by Table 3-1 and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples. EMSL-Ci has also taken the stance that "reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology." Additionally, in written correspondence from the Office of Solid Waste, Oliver Fordham stated " As a "representative sample" can be assured, scaling causes no loss of precision and accuracy in the analysis."

17.1.2 Modifications Specific to Method 3010A

17.1.2.1 Section 11.11.3 of this SOP requires the sample be reduced to a volume of 15 - 20 mL. Section 7.2 of Method 3010A states the volume should be reduced to 3 mL, but also states that no portion of the bottom of the beaker should go dry. The SOP required volume is a closer approximation of the volume required to provide an adequate covering of the beaker so as to prevent the loss of critical analytes through volatilization.

17.1.3 Modifications Specific to MCAWW Methods

17.1.3.1 It was determined by technical review that several of the MCAWW methods were equivalent to the SW-846 methods and therefore were combined under the scope of this SOP as described in Section

11.0. The nature of the differences were deemed insignificant in regards to the amount of acid added and the evaporative volume based on the flexibility allowed by the methods (i.e., add additional acid as required) and the subjective wording of the methods (i.e., evaporate to near dryness vs. an exact volume).

APPENDIX A - TABLES

TABLE I: Approved Preparation Method Analytes - SW846

ELEMENT	Symbol	CAS Number	3005A	3010A
Aluminum	Al	7429-90-5	X	X
Antimony	Sb	7440-36-0	X	
Arsenic	As	7440-38-2	X	X
Barium	Ba	7440-39-3	X	X
Beryllium	Be	7440-41-7	X	X
Cadmium	Cd	7440-43-9	X	X
Calcium	Ca	7440-70-2	X	X
Chromium	Cr	7440-47-3	X	X
Cobalt	Co	7440-48-4	X	X
Copper	Cu	7440-50-8	X	X
Iron	Fe	7439-89-6	X	X
Lead	Pb	7439-92-1	X	X
Magnesium	Mg	7439-95-4	X	X
Manganese	Mn	7439-96-5	X	X
Molybdenum	Mo	7439-98-7	X	X
Nickel	Ni	7440-02-0	X	X
Potassium	K	7440-09-7	X	X
Selenium	Se	7782-49-2	X	X
Silver	Ag	7440-22-4	X	X
Sodium	Na	7440-23-5	X	X
Thallium	Tl	7440-28-0	X	X
Vanadium	V	7440-62-2	X	X
Zinc	Zn	7440-66-6	X	X

X - Designates that the preparation method is approved for an element.

Note: *Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.*

TABLE II: Approved Preparation Method Analytes – NPDES

ELEMENT	Symbol	CAS Number	200.7 (9.4)	200.7 (9.3)
Aluminum	Al	7429-90-5	X	X
Antimony	Sb	7440-36-0	X	X
Arsenic	As	7440-38-2	X	X
Boron	B	7440-42-8	X	X
Barium	Ba	7440-39-3	X	X
Beryllium	Be	7440-41-7	X	X
Cadmium	Cd	7440-43-9	X	X
Calcium	Ca	7440-70-2	X	X
Chromium	Cr	7440-47-3	X	X
Cobalt	Co	7440-48-4	X	X
Copper	Cu	7440-50-8	X	X
Iron	Fe	7439-89-6	X	X
Lead	Pb	7439-92-1	X	X
Magnesium	Mg	7439-95-4	X	X
Manganese	Mn	7439-96-5	X	X
Molybdenum	Mo	7439-98-7	X	X
Nickel	Ni	7440-02-0	X	X
Potassium	K	7440-09-7	X	X
Selenium	Se	7782-49-2	X	X
Silicon	Si	7631-86-9	X	X
Silver	Ag	7440-22-4	X	X
Sodium	Na	7440-23-5	X	X
Thallium	Tl	7440-28-0	X	X
Vanadium	V	7440-62-2	X	X
Zinc	Zn	7440-66-6	X	X

X - Designates that the preparation method is approved for an element

Note: *Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.*

TABLE III: ICP Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	Working Laboratory Control Sample (LCS)/Matrix Spike (MS) Standard (mg/L)	Aqueous Laboratory Control Sample (LCS)/ Matrix Spike (MS) Level * (ug/l)
Aluminum	100	2000
Antimony	25	500
Arsenic	100	2000
Barium	100	2000
Beryllium	2.5	50
Cadmium	2.5	50
Calcium	2500	50000
Chromium	10	200
Cobalt	25	500
Copper	12.5	250
Iron	50	1000
Lead	25	500
Magnesium	2500	50000
Manganese	25	500
Molybdenum	50	1000
Nickel	25	500
Potassium	2500	50000
Selenium	100	2000
Silver	2.5	50
Sodium	2500	50000
Thallium	100	2000
Vanadium	25	500
Zinc	25	500
Boron	50	1000
Tin	100	2000
Titanium	50	1000
Lithium	50	1000
Silicon	50	1000
Strontium	50	1000

* *Levels shown indicate the spike concentration in the final digestate of the aqueous laboratory control sample (LCS) or matrix spike (MS) based on the addition of 1.0 mL working spike (7.3) to 50 mL of sample.*

TABLE IV: ICPMS Aqueous Laboratory Control Sample (LCS) and Matrix Spike/Matrix Spike Duplicate (MS/MSD) Levels

ELEMENT	Working Laboratory Control Sample (LCS)/Matrix Spike (MS) Standard (mg/L)	Aqueous Laboratory Control Sample (LCS)/Matrix Spike (MS) Level* (ug/L)
Aluminum	1000	10000
Antimony	10	100
Arsenic	100	1000
Barium	100	1000
Beryllium	100	1000
Cadmium	100	1000
Calcium	1000	10000
Chromium	100	1000
Cobalt	100	1000
Copper	100	1000
Iron	1000	10000
Lead	100	1000
Magnesium	1000	10000
Manganese	100	1000
Molybdenum	10	100
Nickel	100	1000
Potassium	1000	10000
Selenium	100	1000
Silver	10	100
Sodium	1000	10000
Strontium	100	1000
Thallium	25	250
Vanadium	100	1000
Zinc	100	1000
Boron	10	100
Tin	10	100
Titanium	10	100
Tungsten	10	100

* *Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or MS based on the addition of 0.5 mL working spike (Section 7.4) to 50 mL of sample.*

Note: *Spiking levels may be adjusted as long as the concentrations are documented.*

TABLE V: TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels for ICP

ELEMENT	RL (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)*
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

* *Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or MS based on the addition of 0.5 mL working spike (Section 7.5) to 50 mL of sample.*

APPENDIX B

CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All glassware must be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered Latex Gloves must not be used in the metals laboratory since the powder contains zinc, as well as other metallic analytes. Only unpowdered latex or nitrile gloves must be used in the metals laboratory.

Glassware must be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Canton

SOP No. NC-OP-033, Rev. 4
Effective Date: 10/31/13

Title: TOXICITY CHARACTERISTIC LEACHING PROCEDURE AND SYNTHETIC PRECIPITATION LEACHING PROCEDURE

[Method: SW846 Methods 1311 and 1312]

Approvals (Signature/Date):

Technology Specialist	10/02/13 Date	Health & Safety Coordinator	10/02/13 Date
Quality Assurance Manager	10/31/13 Date	Laboratory Director	10/30/13 Date

This SOP was previously identified as SOP NC-OP-033, Rev 3, dated 8/22/12

Facility Distribution No. _____ **Distributed To: _____**

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LIST OF APPENDICES

1. SCOPE AND APPLICATION

1.1. This SOP describes the application of the Toxicity Characteristic Leaching Procedure (TCLP), SW846 Method 1311. The Toxicity Characteristic (TC) of a waste material is established by determining the levels of 8 metals and 31 organic chemicals in the aqueous leachate of a waste. The TC is one of four criteria in 40 CFR Part 261 to determine whether a solid waste is classified as a hazardous waste. The other three are corrosivity, reactivity, and ignitability. The TC Rule utilizes the TCLP method to generate the leachate under controlled conditions that were designed to simulate leaching through a landfill. EPA's "worst case" waste disposal model assumes mismanaged wastes will be exposed to leaching by the acidic fluids generated in municipal landfills. The EPA model also assumes the acid/base characteristics of the waste will be dominated by the landfill fluids. The TCLP procedure directs the testing laboratory to use a more acidic leaching fluid if the sample is an alkaline waste, again in keeping with the model's assumption that the acid fluids will dominate leaching chemistry over time.

1.2. The specific list of TC analytes and regulatory limits may be found in Appendix A. Other regulatory programs or client specific projects may identify alternative action limits and/or additional constituents of concern.

1.3. This SOP also describes the application of the Synthetic Precipitation Leaching Procedure (SPLP) which was designed to simulate the leaching that would occur if a waste was disposed in a landfill and exposed only to percolating rain water. The procedure is based on SW846 Method 1312. The list of analytes for SPLP may extend beyond the toxicity characteristic compounds shown in Appendix A. With the exception of the use of a modified extraction fluid, the SPLP and TCLP protocols are essentially equivalent. Where slight differences may exist between the SPLP and TCLP, they are distinguished within this SOP.

1.4. The procedure is applicable to liquid, solid, and multiphase wastes.

1.5. The results obtained are highly dependent on the pH of the extracting solution, the length of time that the sample is exposed to the extracting solution, the temperature during extraction, and the particle size/surface area of the sample. These parameters must be carefully controlled.

1.6. The reporting limits are based on the individual samples as well as the individual analysis techniques. However, the sample is determined to be hazardous if it contains any analyte at levels greater than or equal to the regulatory limits.

1.7. If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the procedure need not be run. If the total analysis results indicate that TCLP is not required, the decision to cease TCLP analysis should be remanded to the client.

1.8. Volatile organic analysis of the leachate obtained using a bottle extraction, normally used for extractable organics and metals, can be used to demonstrate that a waste is hazardous, but only the ZHE option can be used to demonstrate that the concentration of volatile organic compounds is below regulatory limits due to potential analyte loss into the headspace during the bottle extraction.

2. SUMMARY OF METHOD

2.1. For liquid wastes that contain less than 0.5% dry solid material, the waste, after filtration through 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP leachate.

2.2. For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solids and stored for later analysis or recombination with the leachate. The particle size of the remaining solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. For TCLP, the extraction fluid employed for extraction of non-volatile analytes is a function of the alkalinity of the solid phase of the waste. For SPLP, the extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. If the sample is a waste or wastewater the extraction fluid employed is a pH 4.2 solution. Two leachates may be generated: a) one for analysis of non-volatile constituents (semi-volatile organics, pesticides, herbicides and metals, and/or, b) one from a Zero Headspace Extractor (ZHE) for analysis of volatile organic constituents. Following extraction, the liquid leachate is separated from the solid phase by filtration through a 0.6 to 0.8 μm fiber filter.

2.3. If compatible (i.e., multiple phases will not form on combination), the initial liquid filtrate of the waste is added to the liquid leachate and these are prepared and analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3. DEFINITIONS

3.1. "Leachate" is used to refer to the solutions generated from these procedures (TCLP, SPLP, deionized water leach).

3.2. "Wet Solids" is that fraction of a waste sample from which no liquid may be forced out by pressure filtration.

3.3. Other definitions of terms used in this SOP may be found in the glossary of the TestAmerica Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Oily wastes may present unusual filtration and drying problems. If requested by the client and as recommended by EPA (see Figure 3), oily wastes can be assumed to be 100% liquid and analysis for total concentrations of contaminants will be performed. This applies

specifically to samples containing viscous non-aqueous liquids that would be difficult to filter. Alternately, the oil may be subjected to pressure filtration. The portion that passes through the filter will be prepared and analyzed separately as an organic waste. The "wet solid" portion that remains behind on the filter will be subjected to leaching--prepared and analyzed. The results will then be mathematically combined.

4.2. Wastes containing free organic liquids (e.g., oil, paint thinner, fuel) usually require dilution prior to analysis to address the matrix interferences. In most instances this results in reporting limits elevated above the TCLP regulatory limits.

4.3. Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks as described in Section 9 and the individual determinative SOPs.

4.4. Glassware and equipment contamination may result in analyte degradation. Soap residue on glassware and equipment may contribute to this. All glassware and equipment should be rinsed very carefully to avoid this problem.

4.5. Phthalates may be eliminated by proper glassware cleanup and by avoiding plastics. Only borosilicate glass, PTFE, or Type 316 stainless steel tumblers may be used for leachates to be analyzed for organics. Plastic tumblers may be used for leachates to be analyzed for the metals.

4.6. Overexposure of the sample to the environment will result in the loss of volatile components.

4.7. Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Gas pressurized equipment is employed in this procedure. Be sure all valves and gauges are operating properly and that none of the equipment, especially tubing, is over-pressurized. CAUTION: Do not open equipment that has been pressurized until it has returned to ambient pressure.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Glacial Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive	2 mg/m^3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.5. A rotary agitation apparatus is used in this procedure. Certain samples may break the glass jars used in the procedure. For these samples, extra caution, including plastic or polyethylene overwraps of the glass jar, may be necessary. Turning the jar or bottle sideways rather than tumbling end over end may also reduce the chance of breakage. If sideways tumbling is used, note this change in the logbook "Comment" section. Guards must be placed in front of any rotating equipment.

5.6. Secure the tumbler and extraction apparatus before starting rotary agitation apparatus.

5.7. During sample rotation, pressure may build up inside the bottle. Periodic venting of the bottle will relieve pressure.

5.8. Any sample requiring cyanide or sulfide analysis should be prepped as a DI leach. When exposed to acidic conditions, the release of hydrogen cyanide gas or hydrogen sulfide gas is possible. SPLP Fluid #3 reagent water must be used for these samples. **NOTE: Do not use an acidic SPLP fluid due to the potential release of hydrogen cyanide gas or hydrogen sulfide gas.**

5.9. Exposure to hazardous chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened

in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.10. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.11. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and Laboratory Supervisor.

5.12. Due to the potential for ignition, flammability or production of noxious fumes, do not attempt to dry non-aqueous liquid samples in an oven. Use extended drying in a ventilation hood.

6. EQUIPMENT AND SUPPLIES

6.1. Extraction vessels

6.1.1. For volatile analytes - zero-headspace extraction (ZHE) vessel, gas-pressure actuated, Associated Design 3745 ZHE or equivalent (see Figure 2)

6.1.2. For metals - either borosilicate glass jars (2.5 L, with Teflon lid inserts) or 2.5 L HDPE (Nalgene or equivalent) bottles may be used

6.1.3. For semi- or non-volatile organics - only borosilicate glass may be used

6.2. Vacuum filtration apparatus and stainless steel pressure filtration apparatus (142 mm diameter), capable of 0 - 50 psi

6.3. Borosilicate glass fiber filters, 0.6 - 0.8 μm (Whatman GF/F 14.2 cm, 9.0 cm, 4.7 cm, 0.7 μm or equivalent): When analyzing for metals, wash the filters with 1 N nitric acid and de-ionized water prior to use, or purchase pre-washed filters. Glass fiber filters are fragile and should be handled with care.

6.4. Rotary agitation apparatus, multiple-vessel, Associated Design and Manufacturing Company 3740-6 or equivalent (see Figure 1): The apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm.

6.5. ZHE Extract Collection Device: Gas-tight syringes, 50 or 100 mL capacity, Hamilton 0158330 or equivalent

6.6. Top loading balance, capable of 0 - 4000 ± 0.01g (all measurements are to be within ± 0.1 grams)

6.7. pH meter and probe capable of reading to the nearest 0.01 unit, and with automatic temperature compensation

6.8. Magnetic stirrer/hotplate and stirring bars

6.9. VOA vials, 40 mL, with caps and septa

6.10. Glass bottles, 1 liter, with Teflon lid-inserts

6.11. Nalgene plastic bottles or equivalent, 1 liter

6.12. Miscellaneous laboratory glassware and equipment

7. REAGENTS AND STANDARDS

7.1. Reagent water for non-volatile constituents must be produced by a Millipore DI system or equivalent. For volatile constituents, water must be passed through an activated carbon filter bed (Milli-Q or tap water passed through activated carbon). Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Hydrochloric acid, 1 N: Carefully add 83 mL concentrated reagent grade HCl to approximately 800 mL reagent water. Cool and dilute to one liter with reagent water. Cap and shake to mix well.

7.3. Sodium hydroxide, 1 N: Carefully add 80g reagent grade NaOH pellets to approximately 1.5 liters of reagent water and stir until dissolved. Cool and dilute to two liters with reagent water.

CAUTION: Heat is generated during this process.

7.4. Sodium hydroxide, 5 N: Carefully add 600g reagent grade NaOH pellets to approximately 2500mL of reagent water and stir until dissolved. Cool and dilute to three liters with reagent water.

CAUTION: Heat is generated during this process.

7.5. Acetic acid, glacial: concentrated, reagent grade liquid (HOAc)

7.6. pH calibration solutions: buffered to a pH of 4, 7, and 2. Commercially available.

7.7. TCLP Leaching Fluids

7.7.1. General Comments

7.7.1.1. The pH probes are to be calibrated prior to use.

7.7.1.2. The leaching fluids MUST be prepared correctly. If the desired pH range is not achieved and maintained, the TCLP may yield erroneous results due to improper leaching. If the pH is not within the specifications, the fluid

must be discarded and fresh extraction fluid prepared. The pH of TCLP fluids #1 and #2 may not be adjusted by the addition of acid or base.

7.7.1.3. Additional volumes of extraction fluids listed below may be prepared by multiplying the amounts of acetic acid and NaOH by the number of liters of extraction fluid required.

7.7.1.4. At the end of the day, all remaining buffer solutions must be properly discarded. The buffer solutions must be used only on the day they were prepared.

7.7.2. TCLP Fluid #1: Carefully add 5.7 mL glacial acetic acid and 64.3 mL of 1 N NaOH to 500 mL reagent water in a 1 liter volumetric flask. Dilute to a final volume of 1 L with reagent water, cap and shake to mix well. For 8 L of fluid use 45.6 mL glacial acetic acid and 514.4 mL 1N NaOH, dilute to 8 L with reagent water. For 40L of fluid, use 228.0 mL of glacial acetic acid and 514.4 mL of 5N NaOH. When correctly prepared, the pH of this solution is 4.93 ± 0.05. The density of TCLP fluid #1 is 0.997 g/mL. If a pH within this range is not achieved, the fluid must be discarded and fresh fluid prepared. The pH may not be adjusted by the addition of additional acid or base.

Note: 514.4mL may need to be determined gravimetrically. 1N NaOH has a density of approximately 1.040g/mL at 23°C and 514.4mL of 1N NaOH has a mass of 534.97g at 23°C. 5M NaOH has a density of approximately 1.21742 g/mL at 23°C and 514.4 mL of 5M NaOH has a mass of 626.24 g at 23°C.

7.7.3. TCLP Fluid #2: Carefully add 5.7 mL glacial acetic acid to 500 mL reagent water in a 1 liter volumetric flask. Dilute to a final volume of 1 L with reagent water, cap and shake to mix well. For 8 L of fluid use 45.6 mL glacial acetic acid, dilute to 8 L with reagent water. When correctly prepared, the pH of this solution is 2.88 ± 0.05. The density of TCLP fluid #2 is 0.997 g/mL. If a pH within this range is not achieved, the fluid must be discarded and fresh fluid prepared. The pH may not be adjusted by the addition of additional acid or base.

7.8. Nitric acid, 50% solution: Slowly and carefully add 500 mL concentrated HNO_3 to 500 mL reagent water. Cap and shake to mix well.

7.9. Sulfuric acid / nitric acid (60/40 weight percent mixture) H_2SO_4/HNO_3: Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid.

7.10. SPLP Leaching fluids

7.10.1. SPLP solutions are unbuffered and exact pH may not be attained. The pH of TCLP and SPLP fluids should be checked prior to use. If not within specifications, the fluid should be discarded and fresh fluid prepared. SPLP fluid pH may be adjusted by the addition of the sulfuric and nitric acids mixture, but may not be adjusted by the addition of base.

7.10.2. SPLP fluid #1: Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 4.20 ± 0.05 This fluid is used for soils from a site that is east of the Mississippi River and for wastes and waste waters. The 60/40 weight percent mixture sulfuric and nitric acids mixture may be diluted by adding several drops of the acid mixture (less than .5mL) to approximately 40mL of reagent water in a vial prior to use in this fluid.

Note: Acid should always be added to water when diluting. Do not add water to acid.

7.10.3. SPLP fluid #2: Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 5.00 ± 0.05. This fluid is used for soils from a site that is west of the Mississippi River. The 60/40 weight percent mixture sulfuric and nitric acids mixture may be diluted by adding several drops of the acid mixture (less than .5mL) to approximately 40mL of reagent water in a vial prior to use in this fluid.

Note: Acid should always be added to water when diluting. Do not add water to acid.

7.10.4. SPLP fluid #3: This fluid is reagent water and is used for leaching of volatiles. Additionally, any cyanide-containing waste or soil is leached with fluid #3 because leaching of cyanide containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

7.11. Methanol and methylene chloride - used to aid in cleaning oil contaminated equipment.

Note: All equipment must be thoroughly washed and rinsed following methylene chloride exposure to eliminate contamination of the ZHE vessels and VOC samples. Only methanol should be stored in the leachate room; any cleaning in which methylene chloride or other solvents is used should be carried out in the Organic Prep room. Equipment that is cleaned with methylene chloride or solvents other than methanol should be washed with soap and water before leaving the Organic Prep room, and again upon its return to the leachate room. This is to prevent solvent contamination of the ZHE process and vessels.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Samples being analyzed for semi- and non-volatile organic compounds should be collected and stored in glass containers with Teflon lid liners. Chemical preservatives shall NOT be added **UNTIL AFTER** leachate generation. Glass containers should be recycled after use.

8.2 Samples being analyzed for metals only can be collected in either glass or polyethylene containers.

8.3 When the waste is to be evaluated for volatile analytes, care should be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon lined septum capped vials with minimal headspace and stored at 4 ± 2°C). Samples should be

opened only immediately prior to extraction.

8.4 Samples should be refrigerated to 4 ± 2°C unless refrigeration results in irreversible physical changes to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

8.5 The minimum TCLP sample collection size is determined by the physical state or states of the waste and the analytes of concern. The amount of waste required varies with the percent solids. The lower the percent solids, the more waste will be required for preliminary and final testing. For aqueous samples containing between 0.5 and 10% solids, several kilograms of sample are required to complete the analyses. The general minimal requirements when the samples are 100% solids include: 1 - 32 oz jar for semi-volatile organic analysis and metals, and 1 - 4 oz jar for volatile organic analysis. Low-density sample materials, such as rags or vegetation, will require larger volumes of sample. For liquid samples (less than 50% solids), the minimum requirements are 2 x 32 oz. jars for semi-volatile organic analysis and metals, and 2 x 8 oz. jars for volatile organic analysis. If volatile organic analysis is the only requested parameter, 2 separate jars are required. If matrix spike or duplicate control samples are requested, additional sample volume is required. If sufficient sample volumes were not received, analyses cannot be started and the client should be notified as soon as possible.

8.6 TCLP leachates should be prepared for analysis and analyzed as soon as possible following extraction. Leachates or portions of leachates for metallic analyte determinations must be acidified with nitric acid to a pH less than 2, unless precipitation occurs. If precipitation occurs upon addition of nitric acid to a small aliquot of the leachate, then the remaining portion of the leachate shall not be acidified and the leachate shall be analyzed as soon as possible. All other leachates should be stored under refrigeration (4 ± 2°C) until analyzed. ZHE leachates must be stored in VOA vials filled to eliminate all headspace.

8.7 Samples are subject to appropriate treatment within the following time periods.

Table 1
Holding Times (Days)

Parameter	Collection to Start of TCLP Leach	End of TCLP Tumble to Preparation	Start of TCLP Leach or Semi-volatile Prep Extraction to Analysis	Total Elapsed Time
Volatiles	14	N/A	14	28
Semi-volatiles	14	7	40	61
Mercury	28	N/A	28	56
Other Metals	180	N/A	180	360

NOTE: The initial holding time is measured from date of collection to date TCLP extraction started. (This should be the TCLP extraction date in LIMS.) Semi-volatile method prep

holding time is measured from the day tumbling is complete to the start of method extraction. Subsequent analysis holding times are measured from the date extraction (TCLP or method prep) starts. If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding holding times is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory limit. The Total Elapsed Time is to be used as guidance. If preps are initiated at the last possible moment of a holding time, the elapsed times may be exceeded.

9. QUALITY CONTROL

9.1. Quality Control Batch (QC Batch)

9.1.1. QA-003 defines a QC Batch as a set of up to 20 field samples of similar matrix that behave similarly and are processed using the same procedures, reagents and standards within the same time period. The same lot of reagents must be used within a batch. A minimum of one TCLP extraction blank (Method Blank), one Laboratory Control Sample (LCS), and one Matrix Spike (MS) will be prepared with each TCLP leachate batch.

9.2. TCLP Extraction Blanks

9.2.1. A minimum of one blank (using the same extraction fluid as used for the samples) must be prepared and analyzed for every batch of samples extracted in a particular vessel type. The blanks are generated in the same way as the samples (i.e., blanks will be tumbled and filtered with the samples). If particle size reduction was performed on any sample in the batch, an equipment blank will be generated by passing blank fluid through the particle reduction apparatus. ZHE Extraction vessels will be uniquely numbered. Consult the TestAmerica QC Program and the individual analysis SOPs for blank acceptance criteria.

9.3. Laboratory Control Sample (LCS)

9.3.1. A LCS is required with each batch of 20 or fewer samples. The LCS shall be generated after a batch of TCLP leachates have been generated (i.e., at the time of the preparative digestion or extraction) by spiking an aliquot of the appropriate extraction fluid used for that batch or reagent water. Consult the individual analysis SOPs for additional LCS guidance (i.e., spike amounts, spike levels, recovery criteria, etc.).

9.4. Matrix Spike (MS)

9.4.1. Matrix spikes are used to monitor the performance of the analytical methods on the matrix and to assess the presence of interferences. An MS is required with each batch of 20 or fewer samples.

9.4.2. Matrix spikes are to be added after filtration of the TCLP leachate. Spikes are not to be added prior to the TCLP leaching. For metals, matrix spikes are to be added before preservation with nitric acid.

9.4.3. Consult the individual analysis SOPs for additional guidance on spike compounds and levels.

9.5. Corrective Actions

9.5.1. Consult the TestAmerica QC Program and individual analysis SOPs for corrective action for blanks and LCS.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to appropriate analysis SOPs.

10.1.1. The calibration of bottle top dispensers and non-Class A beakers is required. Per DoD requirements, a check is performed on these quarterly and the records are maintained electronically.

10.2. Calibrate the pH meter on each day of use in accordance with the electrometric pH SOP NC-WC-010, using fresh buffer solutions. Record the pH meter calibration in the TCLP pH meter calibration electronic logbook.

11. PROCEDURE

11.1. General Comments

11.1.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented on a Nonconformance Memo.

11.1.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.1.3 All masses should be recorded to the nearest 0.1 g.

11.2. Preliminary Sample Evaluations (Refer to Flow Chart #1, Appendix D)

11.2.1 Determine the total volume of TCLP leachate (solid phase leachate plus liquid filtrate) that needs to be generated for analysis according to the following:

Table 2
Minimum Recommended TCLP Leachate Volume

Analysis	Minimum TCLP Required Volume (mL)	Minimum SPLP Required Volume (mL)
Volatiles	2 x 40	2 x 40
Semi-volatiles	250	1000
Pesticides	250	250
Herbicides	100	1000
PCBs	250	250
Metals (Hg and ICP)	100	100
Metals (ICP only)	50	50
Metals (Hg only)	50	50

11.2.1.1. It is suggested to give additional volume for each test as the above values are minimum required volumes for use without dilution.

11.2.1.2. For TCLP and SPLP samples used for matrix spike analysis, at least two times the listed volumes are required. For TCLP and SPLP samples used for both matrix spike and matrix spike duplicate analysis, at least three times the listed volumes are required.

11.2.2 Sample Description (determine sample matrix)

11.2.2.1 Solid - If the waste will obviously yield no free liquid when subjected to pressure filtration, then proceed to Section 11.2.5 or 11.4 (Bottle Extraction Procedure or ZHE Procedure).

11.2.2.2 Liquid - If the sample is a monophasic liquid, proceed to Section 11.2.3 (Percent Solid Determination).

11.2.2.3 Multiphasic– The sample has discernible layers (liquid/liquid or liquid/solid). If more than one container of multiphasic materials is received from the field, each container might show different amounts of each phase. Consult client to determine sample selection alternatives (composite all sample containers, select one, resample, etc.) if this occurs. Multiphasic liquids are also subject to Section 11.2.3 (Percent Solid Determination) if expected to contain less than .5% dry solids.

11.2.3 Solids Determination

11.2.3.1 Determine Type of Filtration Apparatus and Process

11.2.3.1.1 Percent Solids and ZHE Extractions- The ZHE filtration apparatus cannot accurately determine percent solids less than 5%. If an extraction is to be performed solely for volatile organic compounds and the percent solids concentration is apparently greater than 5%, proceed to Section 11.4 (ZHE Extraction Procedure, Volatile Constituents). Otherwise, continue with Section 11.2.3.2. The aliquot of sample used here cannot be used again for the ZHE extraction.

11.2.3.1.2 If the sample is mostly a non-viscous liquid (water or non-viscous organic liquid) of low solids content (expected to be < 0.5%), vacuum filtration should be used initially. Proceed to determination of percent dry solids (Section 11.2.3.2).

11.2.3.1.3 If the sample is viscous (sludge, extremely viscous oil, or is expected to have solids content > 0.5%), use pressure filtration. Proceed to determination of wet solids (Section 11.2.3.3).

11.2.3.2 Determination of Percent Dry Solids

11.2.3.2.1 Measure and record the weight of the filter. Load the filter into the filter holder and assemble vacuum filter apparatus.

11.2.3.2.2 Homogenize the waste; then transfer a subsample of at least 100 g into a clean beaker or snap-top vial. Record the sample weight in the percent dry solids section of the logbook.

11.2.3.2.3 Turn on vacuum source. Transfer the sample to the vacuum filtration device attempting to spread the waste sample evenly over the surface of the filter. Be sure to transfer all significant particulates from the beaker to the filter. Use a reagent water rinse if necessary.

11.2.3.2.4 Once all liquid has been pulled though the filter, remove the filter with the wet solids from the vacuum filtration apparatus.

11.2.3.2.5 Dry the filter and solid phase at 100 ± 20 ° C for approximately 15 minutes.

Note: To avoid potential flash hazards, if the liquid phase is organic (contains solvents and/or oil), the oven should not be used. Prolonged periods of air-drying may be an appropriate replacement for oven-drying.

11.2.3.2.6 Remove the filter from the oven, and allow to cool.

11.2.3.2.7 Weigh and record the dry weight of filter + particulates.

11.2.3.2.8 The percent dry solids will be calculated by the electronic logbook and appear in the column labeled "% Dry Solids".

11.2.3.2.9 If the percent dry solids is ≥ 0.5%, repeat the drying step. Weigh and record the second filter + particulates dry weight. If the two weighings do not agree within 1%, perform additional drying and weighing until successive weighings agree within 1%.

11.2.3.2.10 If the dry solids result is ≥ 0.5%, proceed to Section 11.2.3.3 using a fresh wet portion of the multiphase waste.

11.2.3.2.11 If the percent solids result is less than 0.5%, discard the solid phase. No leaching will be necessary. Filter sufficient sample with either the pressure filtration system or ZHE system as described in Sections 11.3 and 11.4. The filtrate is the TCLP leachate.

11.2.3.3 Determination of Wet Solids

11.2.3.3.1 Assemble the pressure filtration apparatus (use blunt forceps to handle the 0.6 to 0.8 μm filter membrane).

11.2.3.3.2 Homogenize the waste; transfer a minimum of a 100g subsample to the glass beaker. Measure and record the gross weight in the electronic logbook column labeled "Weight of Container + Sample (g)".

11.2.3.3.3 Measure and record the tare weight of the filtrate collection bottle in the electronic logbook column labeled "Weight of Filtrate Bottle (g)".

11.2.3.3.4 Transfer the sample to the filtration device attempting to spread the waste sample evenly over the surface of the filter. Measure and record the tare weight of the empty glass beaker and any residual sample in the electronic logbook column labeled "Weight of Container + Residue (g)".

11.2.3.3.5 The electronic logbook will calculate the net weight of sample used for testing and it will appear in the column labeled "Difference (A - B) Sample Weight (g)".

11.2.3.3.6 Slowly apply gentle pressure of 10 psi to the filtration apparatus. Allow the sample to filter into the pre-weighed filtrate collection bottle until no **SIGNIFICANT** additional liquid has passed through the filter during a two-minute period.

11.2.3.3.7 If necessary, repeat previous step by increasing the pressure in 10 psi increments until a maximum of 50 psi is reached. Stop the filtration when no additional filtrate is generated within a two-minute period.

Note: Some samples will contain liquid material that does not filter (e.g., oil). Do not attempt to filter the sample again by exchanging filters. Viscous oils, or any wastes that do not pass through the filter, are classified by the method as a solid.

11.2.3.3.8 Remove the filtrate collection bottle, weigh and record the gross weight in the electronic logbook column labeled "Weight of the Filtrate bottle + Filtrate (g)".

11.2.3.3.9 The electronic logbook will calculate the net weight of sample aliquot added to the filtration apparatus. This weight will appear in the column labeled "Difference (E - D) Filtrate Weight (g)". This result is used in the calculation of wet solids, which is calculated by the electronic logbook and appears in the column labeled "Difference (C – F) Wet Solids Weight (g)".

11.2.3.3.10 To determine the amount of filtrate, place the exact same type and size container as the filtrate container next to the filtrate. Add water to the exact level as the filtrate container to the empty container. Transfer the water to a graduated cylinder and record the volume. This step will reduce the amount of contamination, which may exist from transferring the filtrate to a graduated cylinder.

11.2.3.3.11 Retain the filtrate for possible recombination with the leachate in Section 11.3.7. Retain the filter and wet solids for the leaching in Section 11.3.

11.2.3.3.12 For multiphase sample preparations, the electronic spreadsheet will calculate the total weight of wet solids. This weight will appear in the column labeled "Difference (C-F) Wet Solids Weight (g)".

11.2.3.3.13 The weight of wet solids that is tumbled should be at least 100g. If this is not feasible due to the limited solids content, matrix or limited volume of the sample, the project manager should be contacted. The client may wish to provide additional sample. If the test proceeds with less than 100g of wet solids, a nonconformance memo indicating the lack of solids should be generated.

11.2.4 Particle-size Reduction for Fluid Selection

11.2.4.1 The subsample used for fluid selection must consist of particles less than approximately 1 mm in diameter (versus the less than 1 cm requirement for the material used for the actual extraction). The method requires a smaller particle size to partially compensate for the shorter duration of contact time with the leachate solution as compared to the full extraction. Inappropriate use of coarser materials could result in the selection of the wrong fluid type.

11.2.4.2 Surface area exclusion - size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram.

11.2.4.3 If the sample contains particles greater than approximately 1 mm in diameter, crush, cut, or grind the solids to the required size.

11.2.4.4 Consult a supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick, etc.).

11.2.5 Determination of Appropriate Extraction Fluid

11.2.5.1 If the solid content is greater than or equal to 0.5%, and if the sample is being analyzed for metals or nonvolatile organic compounds, the type of leaching solution must be determined.

11.2.5.2 Follow times, temperature, and particle size specified in this section as closely as possible. If reaction time between the acid solution and solid waste is too short or too long, the procedure may produce false pH readings.

11.2.5.3 For SPLP, refer to Section 7.10 for fluid selection. Record the fluid type in the logbook.

11.2.5.4 The TCLP leaching fluid for all volatiles is TCLP Fluid #1.

11.2.5.5 TCLP leach fluid determination for non-volatile analytes

11.2.5.5.1 Weigh out a 5.0 ± 0.1 g subsample (less than 1 mm particle size) of the solid phase into a plastic snap-top vial and record in the logbook. Also weigh 5.0 ± 0.1 g of reagent-free sand into a snap-top vial and record this weight in the logbook. This will serve as a temperature blank during heating.

***Note**: If sample quantity is limited, consult supervisor or project manager.*

***Note:** Many multiphase samples have limited solids quantity. In these instances, use a 5g aliquot of the whole sample. Document this difference in the logbook comment section.*

11.2.5.5.2 Add 96.5 ± 1.0 mL of reagent water to each vial, add magnetic stir bar, close lid to the snap-top vial, and stir for 5 minutes.

11.2.5.5.3 Measure and record the pre-test sample pH in the logbook. It is not necessary to record the pH of the temperature blank.

Note: To avoid damaging a glass pH probe when organic liquid is present, use narrow range pH indicator paper or an ISFET pH meter.

11.2.5.5.4 If the pH is less than or equal to 5, use TCLP Fluid #1.

11.2.5.5.5 If the pH is greater than 5, add 3.5 mL 1 N HCl. Slurry the sample briefly. Insert a thermometer into the temperature blank vial. One temperature blank must be used for each group of samples heated. All samples and the temperature blank in the group must be heated at the same time in order for the temperature of the blank to represent the others. Heat to 50 ± 2°C and maintain for ten minutes.

Note: The heating cycle is a critical step. If the solid waste does not remain in contact with the acidic solution under specified time and temperature conditions, an erroneous pH may be measured.

11.2.5.5.6 Cool to 23 ± 2°C.

11.2.5.5.7 Measure and record the pH immediately after the sample has reached 23 ± 2°C.

11.2.5.5.7.1 If the pH is less than or equal to 5, use TCLP Fluid #1. Record the buffer in the logbook.

11.2.5.5.7.2 If the pH is greater than 5, use TCLP Fluid #2. Record the buffer in the logbook.

11.2.5.5.7.3 If the pH is near 5 and cannot be determined using pH paper, use of the pH meter is required. Record the pH to the proper significant figures as given by the pH meter, if used.

11.2.6 For samples requiring analysis for semi-volatile organics, pesticides, herbicides or metals proceed to Section 11.3.

11.2.7 For samples requiring analysis for volatile organics (ZHE), proceed to Section 11.4.

11.3 Bottle Extraction Procedure: Non-Volatile Constituents: Semi-Volatiles, Pesticides, Herbicides, Metals (Refer to Flow Chart 2, Appendix D).

11.3.1 Evaluate the solid portion of the waste for particle size. If it contains particles greater than 1 cm in size, prepare the solid portion of the waste for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size (i.e., capable of passing through a 9.5 mm, 0.375 inch, standard sieve). Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram. If particle size reduction was required, record this in comments column in logbook.

11.3.1.1 Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick, etc.). Scissors or shears may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Determination of particle size reduction tools should take into account the requested analytes (e.g., avoid chromium steel tools when TCLP metals have been requested). Bricks, rocks, or other solids amenable to grinding may be subcontracted out for particle size reduction (contact PA or PM). Note that size reduction to fine powder is not appropriate, and could invalidate results. If necessary, consult client for guidance.

11.3.2 Determine the minimum total volume of solid phase leachate that needs to be generated. Refer to Section 11.2.1.

11.3.3 Use 100 g of solid unless sample quantity is limited. If limited sample, divide the total volume of solid phase leachate required by 20 to determine the minimum mass of solid phase required for leaching. Round this mass **up** to the nearest 5 g. Client must be notified, and a nonconformance memo issued if less than 100 g of solid material is used. The client may choose to send additional sample volume if insufficient volume is available to meet method requirements of 100g. If additional sample volume is to be received, the TCLP preparation should be stopped until the additional sample arrives or the project manager gives additional direction.

Note: Solid phase material is often in limited quantity from multiphase samples. Generally all the *solid* phase material and the filter from Section 11.2.3.3.11 are transferred to the leaching bottle. The mass of the solid of a multiphase sample is located in the electronic logbook column labeled "Difference (C-F) Wet Solids Weight (g)".

11.3.4 All non-ZHE extraction vessels should be labeled with the sample name on both lid and bottle during weighing and tumbling. Before each tumbling, a new label is to be placed prominently on each reusable glass vessel for each use. After three uses, (once the bottle has three labels) the bottle is to be discarded. If the vessel cracks, breaks, becomes scratched, or is used for an oily sample, it is to be discarded immediately after use.

11.3.5 Weigh the required mass of solid phase into an appropriate extraction vessel (plastic for metals only, borosilicate glass for all others), and **slowly** add 20 times its mass of appropriate leaching fluid (e.g., 100 g of sample would require 2000 mL of leaching fluid). Record the weight of the sample aliquoted for the extraction. Record the volume of extraction fluid added in the logbook if other than 2000 mL. This volume is calculated by the electronic logbook for multiphase samples; the result can be found in the column labeled "G x 20 Buffer Amount (mL)".

11.3.6 Ensure any effervescence has stopped before capping the bottle tightly. Secure in a rotary agitator and rotate end-over-end at 28-32 rpm for 16-20 hours. The temperature of the room should be 23 ± 2°C. The temperature of the room is recorded using a continuous temperature monitor. Record the rotary agitator I.D. and the date and time extraction is started and completed in the logbook. Record the RPM of the agitator(s) in the LIMS batch in the "Batch Information" window.

Note: As agitation continues, pressure may build up within the bottle for some types of wastes. To relieve excessive pressure, the bottle may be removed and opened periodically in a properly vented hood to relieve any built-up pressure.

11.3.7 After tumbling in the rotary agitator is completed, remove the bottle and allow the solids to settle. Record the date and time the extraction is completed in the logbook. If sample was multiphase with an initial filtrate, drop a few drops of the filtrate (with a disposable glass pipette) into the extraction bottle and observe whether the filtrate is insoluble or forms a precipitate with the leachate. If so then the filtrate is not compatible with the leachate and must be bottled and analyzed separately. The results are normally mathematically recombined (Section 12.1.2). If the filtrate is compatible with the leachate (i.e., completely soluble) then pour the entire filtrate into the leachate bottle, recap and mix. Proceed with the leachate filtration step in the next section.

11.3.8 Filter the sample using pressure filtration by filtering through a new glass fiber filter. For final filtration of the TCLP leachate, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filters must be acid washed if metals are to be determined (see Section 6.3). The entire sample need not be filtered; however, sufficient volume should be generated to support the required analyses.

11.3.9 Measure the pH of the TCLP leachate and record in the logbook. A small aliquot (approximately 10 to 15 mL) of the filtered leachate in a separate container should be used to measure the pH of the leachate. The pH probe or paper should not be placed directly into bottles of samples that will be used for further prep or analysis.

11.3.9.1 In general, the use of pH paper is appropriate for determining the final pH of the leachate. The pH meter may be used if it is not possible to determine the pH with pH paper (e.g. there is color interference when using pH paper).

11.3.10 Prepare sufficient volume for MS/MSD quality control testing, as necessary. Refer to the appropriate determinative SOPs for further guidance on the spike components, levels and action criteria.

11.3.11 Immediately preserve the leachate as follows:

Metals	pH < 2 w/ HNO_3 for aqueous filtrates and leachates (do not acidify oils and other non-aqueous liquids)
All others	Refrigerate to 4 ± 2 °C

Note: Refer to Section 8.5 if precipitation occurs upon preservation.

Note: Preservation of Metals samples is generally performed by Metals personnel. The MS/MSD volume must be spiked prior to preservation.

11.3.12 Label each sample with the appropriate information and submit to the appropriate analytical groups for prep and analysis. For multiphase samples requiring mathematical recombination, the electronic logbooks are available to the sample preparation and analysis groups and are attached to the LIMS batches. Most mathematically recombined samples will require data entry for the filtrate and leachate portions as well as for the mathematically recombined results. Contact the Project Manager to ensure the proper sample login is completed.

11.4 ZHE Extraction Procedure: Volatile Constituents (Refer to Flow Chart #3, Appendix D)

11.4.1 Use the ZHE device to obtain a TCLP leachate for analysis of volatile compounds only. Leachate resulting from the use of the ZHE shall NOT be used to evaluate the mobility of non-volatile analytes (e.g., metals, pesticides, herbicides and semi-volatile organics).

11.4.2 Due to some shortcomings of the method, losses of volatile compounds may occur. Extra care should be observed during the ZHE procedure to ensure that such losses are minimized. Charge the ZHE with sample only once and do not open the device until the final extract has been collected. Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere any longer than necessary.

11.4.3 Install new O-rings and adjust the ZHE piston in the ZHE body to the appropriate height (slightly moisten the O-rings with leaching fluid if necessary).

11.4.4 If the preliminary evaluations indicated the need for particle size reduction, homogenize the waste, weigh out a sufficient size subsample and prepare for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size as measured with a ruler (Do NOT sieve the sample). Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram. If particle size reduction was required, record this in the "Comments" column of the logbook.

Note: To minimize loss of volatiles, samples for volatiles that require particle size reduction should be kept in sample storage (at 4°C) until immediately before size reduction. Aggressive reduction, which would generate heat, should be avoided; and exposure of the waste to the atmosphere should be avoided to the extent possible. Size reduction to a fine powder is not appropriate. Also see Section 11.2.

11.4.4.1 Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick). Scissors or shears may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Bricks, rocks, or other solids amenable to grinding may be subcontracted out for particle size reduction (contact PM).

11.4.5 Homogenize and transfer an appropriate size subsample of the waste into the ZHE and record the mass in the logbook.

11.4.5.1 For wastes that are solid, a 25g sample is used, unless sample volume is limited. Smaller weights may also be used if the sample is of an unusual matrix that may damage the ZHE vessel.

11.4.5.2 For wastes containing < 0.5% solids, the liquid portion of the waste, after filtration, is defined as the TCLP leachate. Filter enough of the sample to support all of the volatile analyses required.

11.4.5.3 If the sample has ≥ 0.5% solids and has non-volatile TCLP/SPLP requested, the appropriate sample size should be estimated based on the wet solids content determined in Section 11.2.3.3. If ZHE only, use visual wet solids estimate to sample sub-aliquot. Record the weight of the waste and the beaker in the electronic logbook column labeled "Weight of Container + Sample (g)". After adding the sample to the ZHE extraction vessel, record the weight of empty container and any residue in the electronic logbook column labeled "Weight of Container + Residue (g)". The electronic logbook will calculate the sample weight and the result will appear in the column labeled "Difference (A – B) Sample Weight (g)".

11.4.5.4 The weight of wet solids that tumbles in the ZHE does not need to be 25g. Sufficient leachate should be generated to perform the analysis, however. If there is insufficient leachate volume available after performing the multiphase procedure (including all compatible filtrate that will be returned to the vessel after leaching), it should be started over and all sample volumes generated from the failed procedure should be discarded. **Note:** For wastes containing greater than 0.5% wet or dry solids, the "solids" value from the ZHE filtration process is used to determine the volume of fluid to load into the ZHE. This value is calculated by the electronic spreadsheet and the volume of fluid appears in the column labeled "G x 20 Buffer Amount (mL) Leachate Vol".

11.4.6 Carefully place the glass fiber filter between the support screens and secure to the ZHE. Tighten all the fittings.

11.4.7 Place the ZHE in a vertical position; open both the gas **AND** liquid inlet/outlet valves. Attach a gas line to the gas inlet/outlet valve.

11.4.8 If the waste is solid, slowly increase the pressure to a maximum of 50 psi to force out as much headspace as possible and proceed to Section 11.3.13.

11.4.9 If this is a multiphase sample, carefully apply gentle pressure of 10 psi (or more, if necessary) to force all headspace slowly out of the ZHE. At the **FIRST** appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue gas pressure.

11.4.10 Assemble a syringe and place the plunger in all the way. Attach the pre-weighed syringe to the liquid inlet/outlet valve and open the valve. Record the tare weight of the collection syringe in the electronic logbook column labeled "Weight of Syringe (or vials) (g)". If the volume of the filtrate is expected to exceed the volume of the syringe (e.g., more than 50mL), then record the tare weight of an appropriate number of vials, instead of the syringe.

11.4.11 Carefully apply gas pressure of no more than 10 psi to force out the liquid phase. Allow the sample to filter until no **SIGNIFICANT** additional filtrate has passed in a two-minute period.

Note: If the capacity of the syringe is reached, close the liquid inlet/outlet valve, discontinue gas pressure, remove the syringe, dispense the volume into the pre-weighed vials, record weight in Column E and filtrate volume in the logbook.

11.4.12 Repeat previous step increasing the pressure in 10 psi increments until 50 psi is reached and no significant liquid has passed in a two-minute period. Close the valve and discontinue gas pressure. Remove the collection device and record the total weight of the collection device with filtrate in the electronic logbook column labeled Weight of the Syringe (or vials) + Filtrate (g), and record the filtrate volume in the electronic logbook column labeled "Volume of Filtrate* (mL)." Transfer the filtrate to VOA vials and label appropriately. The weight of the filtrate is calculated by the logbook and appears in the column labeled "Difference (E – D) Filtrate Weight (g)".

Note: If the original waste contained less than 0.5% solids (Section 11.2.3.2), this filtrate is defined as the TCLP leachate and you may proceed to Section 11.3.22. Otherwise, save the vials by storing at 4? C under minimal headspace conditions, for recombination as in Section 11.3.21.

The material remaining in the ZHE is defined to be the "solid" phase. The weight of the solid phase is calculated by the logbook and appears in the column labeled "Difference (C – F) Wet Solids Weight (g)".

11.4.13 Determine the amount of buffer to use. Solid samples of 25g use 500 mL of leach fluid (20 X 25 g). For multiphase samples or other samples using weights of other than 25g, the volume of buffer will be equal to 20 times the weight of the sample. The multiphase sheet of the electronic logbook calculates the volume of

fluid required for multiphase samples and this value appears in the column labeled "G x 20 Buffer Amount (mL) Leachate Volume".

Note: The TCLP ZHE prep uses only TCLP fluid #1; the SPLP ZHE prep uses only SPLP fluid #3 reagent water.

11.4.14 Load the fluid transfer reservoir with an excess of Fluid #1, and preflush the transfer line to eliminate air pockets. Be sure the required volume remains.

11.4.15 Attach the transfer line to the liquid inlet/outlet valve and open the valve. Carefully pump the required volume into the ZHE and close the valve. Disconnect the transfer line.

11.4.16 Check the ZHE to make sure all the valves are closed and manually rotate the ZHE (end-over-end) two or three times. Reposition the ZHE in the vertical position.

11.4.17 Pressurize the ZHE to 5-10 psi. If the ZHE appears to be leaking, follow the corrective action protocols recommended by the manufacturer and repeat the procedure.

11.4.18 Slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced during the introduction of the Fluid. Upon the first sign of liquid from the valve, close the valve.

11.4.19 Repressurize the ZHE to 5-10 psi and place in the rotary agitator. Rotate at 28-32 rpm for 16-20 hours. Record the rpm of the agitator and the date and time of the start and stop of agitation in the appropriate logbooks. Room temperature should be 23 ± 2 °C. The room temperature is recorded using a continuous temperature monitor.

11.4.20 Confirm that the pressure of 5-10 psi was maintained throughout the leaching. If it was **NOT** maintained, return to Section 11.3, and repeat the leaching with a new aliquot of sample.

11.4.21 If there is an initial liquid filtrate (Section 11.3.12) determine if it is compatible with the leachate if the filtrate has not been previously tested (Section 11.3.7).

11.4.21.1 Remove the plunger from the syringe and attach the barrel to the ZHE vessel. Open the outlet valve and pressurize as necessary to transfer about 1 mL of leachate into the syringe. Close the outlet valve.

11.4.21.2 With a glass pipette transfer a few drops of initial filtrate into the open syringe barrel. Formation of separate layers or a precipitate indicates the filtrate and leachate are not compatible. Bottle the filtrate for separate preparation and analysis. The results are normally mathematically recombined.

11.4.21.3 If the filtrate is compatible gently pour the remainder of the filtrate into the syringe barrel. Install the plunger. Bleed any pressure in the ZHE piston. Open the inlet/outlet valve and depress the syringe plunger to inject the filtrate into the ZHE vessel. Do not inject the air bubble (if present) from the syringe.

11.4.21.4 Close the valve and rotate a few times to mix. Proceed with leachate filtration as described in the next section.

11.4.22 Attach an empty syringe to the outlet valve. Open the valve and pressurize the piston to expel the leachate from the ZHE vessel. Following collection, store the TCLP leachate in 2 or 3 40-mL VOA vials with minimal headspace at 4 ± 2 °C and prepare for analysis as soon as possible using the appropriate organic analysis procedure (see Section 16.1).

11.4.23 If the individual phases are analyzed separately, combine the results mathematically by using the recombination calculation in Section 12.1.2. Electronic copies of the TCLP preparation logbook sheets are available to the sample preparation and analysis groups. Most mathematically recombined samples will require data entry for the filtrate and leachate portions as well as for the mathematically recombined results. Contact the Project Manager to ensure the proper sample login is completed.

11.4.24 ZHE Vessel Cleaning

11.4.24.1 Disassemble the vessel.

11.4.24.2 Clean all parts (vessel, lid, bottom, piston, and metal filters) with soapy water.

11.4.24.3 Rinse all parts with tap water followed by DI water.

11.4.24.4 Discard all used gaskets.

12 DATA ANALYSIS AND CALCULATIONS

12.1 Calculations

12.1.1 Calculation of weight of extraction fluid to use:

$$Volume\ of\ extraction\ fluid = 20\ X\ weight\ of\ wet\ solids\ to\ be\ extracted$$

12.1.2 Mathematical recombination of analytical results:

$$Final\ Analyte\ Concentration = \frac{(V_1 \times C_1) + (V_2 \times C_2)}{V_1 + V_2}$$

Where,
V_1 = total volume of the initial filtrate phase (L).
C_1 = analyte concentration in initial filtrate phase (mg/L).
V_2 = volume of the theoretical solid phase leachate (L).
C_2 = analyte concentration in solid phase leachate (mg/L).

12.2 Reporting Requirements

12.2.1 Follow these reporting conventions for multi-phase samples.

12.2.1.1 If both phases have positive results, use the values from each phase to calculate the recombined result. Use the reporting limit for each phase to calculate the recombined reporting limit.

12.2.1.2 If both phases are "ND" (not detected) the recombined result is "ND," and the reporting limit is calculated from the reporting limit for each phase.

12.2.1.3 If one phase is "ND" and the other phase has a positive result, use the zero for the "ND" phase and the positive value for the other phase to calculate the combined result. This will produce a minimum known concentration. Alternatively, at client request, the maximum possible concentration can be calculated by using the reporting limit for the "ND" phase rather than zero. The combined reporting limit is based on the reporting limit for both phases.

12.2.2 Units - regardless of the nature of the sample, all TCLP and SPLP results are reported in units of mg/L.

12.2.3 For limits and significant figures, consult the appropriate analytical methods (Section 16.1).

12.2.4 Anomalies - all anomalies observed during the leach procedure must be noted on the worksheet or an NCM form. Some examples of such anomalies are:

12.2.4.1 Sample was monolithic - particle size reduction not possible due to nature of matrix.

12.2.4.2 Insufficient sample -less than the required 100g minimum was available.

12.3 Review Requirements

12.3.1 Review all applicable holding times. If a holding time was exceeded, confirm that a holding time violation was properly documented in an NCM.

12.3.2 If Total analysis results are available, those results may be compared with the TCLP analysis results according to the following:

$$Total \geq 20 \times TCLP$$

Note: Assumes the sample is 100% Solids.

12.3.3 Total constituent analysis results can be used to demonstrate the TCLP protocol is unnecessary. In performing a TCLP analysis, there is a 20:1 dilution of the original sample with the leaching solution. Thus, if the "total constituent" result is less than 20 times the TC level, it is impossible for the leachate to "fail" and the TCLP does not need to be performed. For example, the TC level for lead is 5.0 mg/L (ppm). Therefore, if a sample of lead-contaminated soil contains less than 100 ppm total lead, a TCLP test need not be run to demonstrate that lead is less than the TCLP limit.

13 METHOD PERFORMANCE

13.1 Refer to individual analysis SOPs.

13.2 Training Qualification

13.2.1 The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14 POLLUTION PREVENTION

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15 WASTE MANAGEMENT

15.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2 The following waste streams are produced when this method is carried out.

15.2.1 Acidic waste from sample extract: This waste is collected in the laboratory in a designated container identified as "Acid Waste".

15.2.2 Buffer solutions: This waste can be poured down the drain with copious amounts of water.

15.2.3 Solid waste from sample extract, solid sample waste and used filter paper from the sample filtration step: This waste is disposed of in a designated container identified as "Solid Waste".

15.2.4 Flammable solvent waste and remaining TCLP extracts: This waste is disposed of in a flammable liquid solvent container identified as "Mixed Flammable Solvent Waste".

15.2.5 Glassware contaminated with acidic sample residue: Broken or unusable glassware is disposed of in a designated container identified as "Solid Waste". Glassware used in this test method is washed per the Glassware Washing SOP.

16 REFERENCES

16.1 References

16.1.1 Method 1311, Toxicity Characteristic Leaching Procedure, Revision 0, July 1992, SW-846 Final Update I

16.1.2 Method 1312, Synthetic Precipitation Leaching Procedure, Revision 0, November 1994, SW-846 Update II

16.1.3 Toxicity Characteristic: Corrections to Final Rule. Method 1311, Federal Register, Vol. 55, No. 126, Friday, June 29, 1990

16.1.4 Toxicity Characteristic: Final Rule. Method 1311, Federal Register, Vol. 55, No. 61, Thursday, March 29, 1990

16.1.5 Technical Background Document and Response to Comments, Method 1311, Toxicity Characteristic Leaching Procedure, USEPA/OSW, April 1989

16.1.6 Corporate Quality Management Plan (CQMP), current version

16.1.7 TestAmerica Canton Quality Assurance Manual (QAM), current version

16.1.8 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

16.1.9 Revision History

Historical File:		Revision 1.0: 02/01/00		Revision 3: 08/22/12
(formerly CORP-IP-004NC)		Revision 1.1: 10/10/00		
		Revision 1.2: 11/11/04		
		Revision 0: 03/31/08 (NC-OP-033)		
		Revision 1: 07/15/10 (NC-OP-033)		
		Revision 2: 6/16/11		

16.2 Associated SOPs and Policies, current version

16.2.1 TestAmerica Canton Quality Control Program, QA-003

16.2.2 Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods, NC-IP-011

16.2.3 Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis, Method 6010B and Method 200.7, NC-MT-012

16.2.4 Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW-846 7470A and MCAWW 245.1, NC-MT-013

16.2.5 Determination of Volatile Organics by GC/MS based on Methods 8260B, NC-MS-019

16.2.6 SGC/MS Analysis Based on Method 8270C, NC-MS-018

16.2.7 Gas Chromatographic Analysis Based on Methods 8000B, 8021B, 8081A, 8082, 8151A, and 8015B, NC-GC-038

16.2.8 Extraction Procedure for Chlorinated Acid Herbicides Based on Method 8151A, NC-OP-031

16.2.9 Continuous Liquid / Liquid Extraction of Organic Compounds from Waters Based on Method SW846 3520C NC-OP-037

16.2.10 Separatory Funnel Extraction of Organic Compounds from Waters Based on Method SW846 3510C and 600 Series NC-OP-038

16.2.11 pH Electrometric Method, NC-WC-010

16.2.12 Supplemental Practices for DoD Project Work, NC-QA-016

16.2.13 Standards and Reagents, NC-QA-017

16.2.14 pH Electrometric Method, NC-WC-010

16.2.15 Glassware Washing, NC-QA-014

16.2.16 Subsampling, NC-OP-046

16.2.17 Soil Processing, NC-OP-044

17 MISCELLANEOUS

17.1 Modifications/Interpretations from Reference Methods

17.1.1 Section 11.2: Preliminary Evaluations. Section 7.1 of the source Method 1311 states that the sample aliquot used for the preliminary evaluation "...may not actually undergo TCLP extraction." Section 7.1.5 of the source method indicates that the portion used for the preliminary evaluation may be used for either the ZHE or non-volatile extraction if the sample was 100% solid. Section 7.1.5 of the source method further indicates that if the sample was subjected to filtration (i.e., < 100% solid) that this aliquot may be used for the non-volatile extraction procedure only as long as sufficient sample is available (minimum 100 g). Samples that have been subjected to the oven-drying step may not be used for TCLP extraction because solid phase degradation may result upon heating.

17.1.2 Sections 11.3.7 and 11.4.21: Determination of Filtrate/Extraction Fluid Compatibility. Section 7.2.13 of the source method provides no guidance as to how to make this determination. As a result, the procedure herein was developed.

17.1.3 Section 9.2: TCLP Extraction Blanks. Section 8.1 of the source method states that a minimum of one blank for every 20 extractions "...that have been conducted in an extraction vessel." TestAmerica has interpreted this to mean one blank per twenty samples leached per TYPE of leaching vessel (i.e., Bottle or ZHE) per leach fluid used.

17.1.4 Section 11.2.5.5.7: Determination of Appropriate Extraction Fluid. Method 1311 does not address the appropriate approach to take if the pH equals 5.0. This SOP requires that Fluid #1 must be used if the pH is less than or equal to 5.0.

17.1.5 Section 9.4: QA/QC - Matrix Spike : Section 8.2 of the source method states "A matrix spike shall be performed for each waste type..." and "A minimum of one matrix spike must be analyzed for each analytical batch". Further, Section 8.2.3 of the source method also states "The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist". The standard TestAmerica QAM is designed to address the performance monitoring of analytical methodology through the LCS program. A minimum of one MS will be prepared for each TCLP leachate batch. The MS results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, the MS results have immediate bearing only on the specific sample spiked and not all samples in the batch.

17.1.6 Section 8.2.2 of the source method states that "In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level". The method also states "If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration but may not be less than five times the method detection limit". For several analytes, spiking at the regulatory level is inappropriate to the range of analysis afforded by the determinative methods. Due to the wide range in these levels, TestAmerica spikes at the levels specified in the determinative SOPs.

APPENDIX A: Tables

Table 3 - Toxicity Characteristic Analytes and Regulatory Levels (Final Rule)	
Contaminant	**mg/L**
Arsenic	5.0
Barium	100.0
Benzene	0.5
Cadmium	1.0
Carbon tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
o-Cresols	200.0
m-Cresols	200.0
p-Cresols	200.0
Total Cresols (used if isomers not resolved)	200.0
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
2,4-Dinitrotoluene	0.13
1,1-Dichloroethylene	0.7
Endrin	0.02
Heptachlor(& epoxide)	0.008
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.5
Hexachloroethane	3.0
Lead	5.0
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
Selenium	1.0
Silver	5.0
Tetrachloroethylene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl chloride	0.2

APPENDIX B: Figures

Figures 1 and 2 - Rotary Agitation Apparatus and Zero Headspace Extraction Vessel (ZHE)

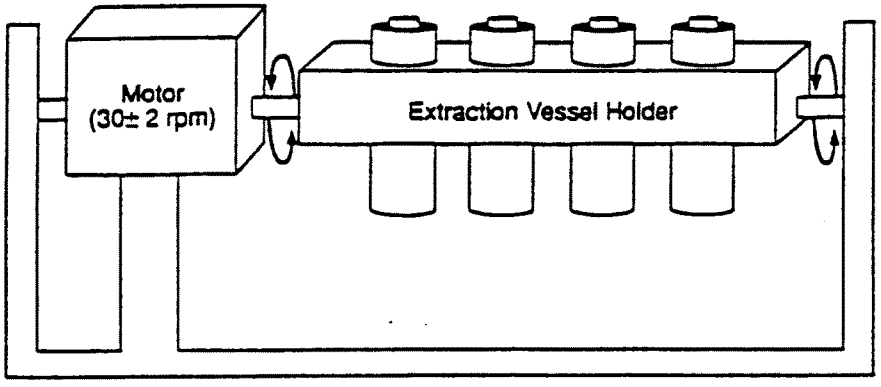

Figure 1. Rotary Agitation Apparatus

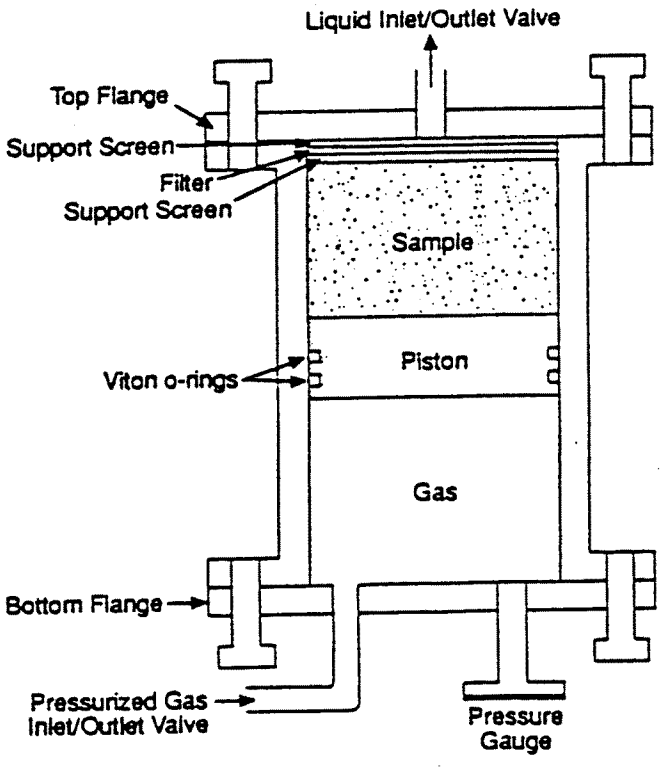

Figure 2 – Cross Section of Zero Headspace Extraction Vessel (ZHE)

Figure 3 - US Environmental Protection Agency Memorandum #35, Page 1

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE

MEMORANDUM # 35

DATE: June 12, 1992

SUBJECT: Notes on RCRA Methods and QA Activities

From: Gail Hansen, Chief
Methods Section (OS-331)

This memo addresses the following topics:

- o 1992 Symposium on Waste Testing and Quality Assurance
- o SW-846 Update
  - Final Rule for January 23, 1989 Proposed Rule
  - Notice, Proposed Rulemaking for the Second Update to the Third Edition
- o Chlorofluorocarbon 113 (CFC-113) Solvent Replacement Update
- o Environmental Monitoring Methods Index (EMMI)
- o Sampling Work Group Formation
- o MICE Update
- o Oily Waste Analysis
- o Electronic SW-846 Availability.

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Figure 3 - US Environmental Protection Agency Memorandum #35, Page 10 (cont'd)

Oily Waste Analysis

One of the most frequently asked questions on the MICE Service concerns the application of the TCLP, Method 1311, to oily wastes. Many callers request technical guidance on the extraction of oily wastes due to the difficulty in the filtration on these types of waste. In many cases, an oily waste does not filter completely due to premature clogging of the glass fiber filter. This can result in the retantion of standing liquid on the glass fiber filter. Material that do not pass through the glass fiber filter at the conclusion of the filtration step is defined by the method as the solid phase of the waste. The solid phase is then subjected to the leaching procedure of the TCLP. For oily wastes, clogging of the glass fiber filter can result in an overestimation of the amount of solid material available for leaching.

To solve this problem, the Agency recommends a conservative approach, one that probably will overestimate the amount of leaching. Rather than performing the TCLP extraction on the unfiltered portion of the oily waste, assume the waste is 100% liquid (e.g., will pass through the glass fiber filter) and perform a totals analysis on the oily waste to determine if the oil exceeds the appropriate regulatory level.

Filterable waste oil generated during the TCLP must be analyzed for a variety of organic and inorganic analytes. The OSW recognizes the difficulty in achieving acceptable performance for the analysis of waste oil using methods currently provided in SW-846. As a result, the Agency will provide several new methods for the preparation and analysis of oil samples to the Organic Methods Workgroup in July. In addition, a microwave assisted digestion procedure should improve the analysis of metals and will be proposed as part of the Second Update of the Third Edition of SW-846. Brief descriptions of these techniques are provided below, for additional information on the organic procedures contact Barry Lesnik at (202) 260-7459. For additional information on microwave digestion contact Ollie Fordham (202) 260-4778.

The use of purge-and-trap (Method 5030) for volatiles in oil generally results in severe contamination of analytical instrumentation. Traps, transfer lines and chromatography columns may become contaminated with oil. This leads to elevated baselines, hydrocarbon background in subsequent analyses, and cross-contamination. Headspace (Method 3810) is currently allowed only as a screening procedure in SW-846. The Agency is evaluating the use of headspace in conjunction with isotope dilution mass spectrometry for the quantitative analysis of volatiles in oil. Headspace reduces interference problems encountered with purge-and-trap. However, headspace quantitation can be questionable because the distribution of analytes is not

APPENDIX C : Logbook Sheets

TESTAMERICA NORTH CANTON ZHE LOGBOOK

Batch #	Sample ID	Ext. Vessel ID	Matrix	Sample Wt (g)	Ext. Buffer ID	Date On	Time On	Initials On	Date Off	Time Off	Initials Off	Tumbler ID	Tumbler Speed 30±2 RPM	Ext. Fluid ID	Initial ZHE Pressure	Final ZHE Pressure	Comments

TESTAMERICA NORTH CANTON ZHE LOGBOOK - MULTIPHASE

	A	B	C	D	E	F			G	H				
Sample Number	Weight of Container + Sample (g)	Weight of Container + Residue (g)	Difference (A – B) Sample Weight (g)	Weight of the Syringe (or vials) (g)	Weight of the Syringe (or vials) + Filtrate (g)	Difference (E – D) Filtrate Weight (g)	Volume of Incompatible Filtrate (mL) (ie, oil)	Volume of Compatible Filtrate (mL) (ie, water filtrate)	Difference (C – F) Wet Solids Weight (g)	G x 20 Buffer Amount (mL) Leachate Vol.	Vessel ID	Initials	Total Water-Soluble Volume in L (TCLP)	Incompatible Volume in L **(Waste Dilution)**

TESTAMERICA NORTH CANTON TCLP LOGBOOK

				Pre-Test			% Dry Solids				Leach Start						Leach Finish					
Batch #	Sample ID	Parameters	Matrix	Wt (g) Sample	pH after water	pH after heating	Wt (g) Sample	Wt (g) Filter	Filter + Particulates Wt (g)	% Dry Solids	Wt (g) Sample	Buffer #	Date On	Buffer ID	Time On	Initials On	Date Off	Time Off	pH Off	Initials Off	Tumbler ID	Comments

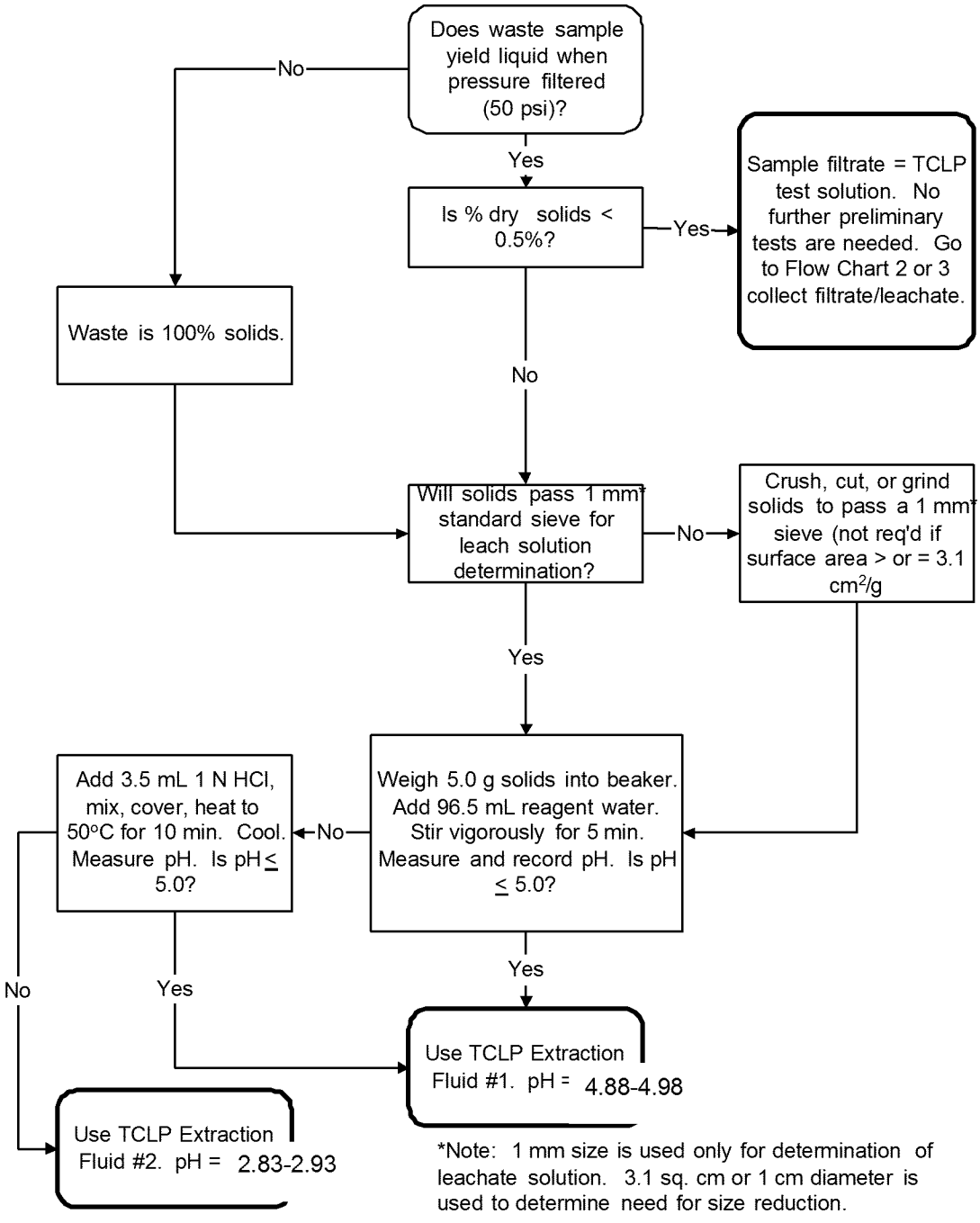
TESTAMERICA NORTH CANTON TCLP LOGBOOK – MULTIPHASE

	A	B	C	D	E	F			G	H				
Sample Number	Weight of Container + Sample (g)	Weight of Container + Residue (g)	Difference (A – B) Sample Weight (g)	Weight of the Syringe (or vials) (g)	Weight of the Syringe (or vials) + Filtrate (g)	Difference (E-D) Filtrate Weight (g)	Volume of Incompatible Filtrate (mL) (ie, oil)	Volume of Compatible Filtrate (mL) (ie, water filtrate)	Difference (C-F) Wet Solids Weight (g)	G x 20 Buffer Amount (mL) Leachate Vol.	Vessel ID	Initials	Total Water-Soluble Volume in L (TCLP)	Incompatible Volume in L (Waste Dilution)

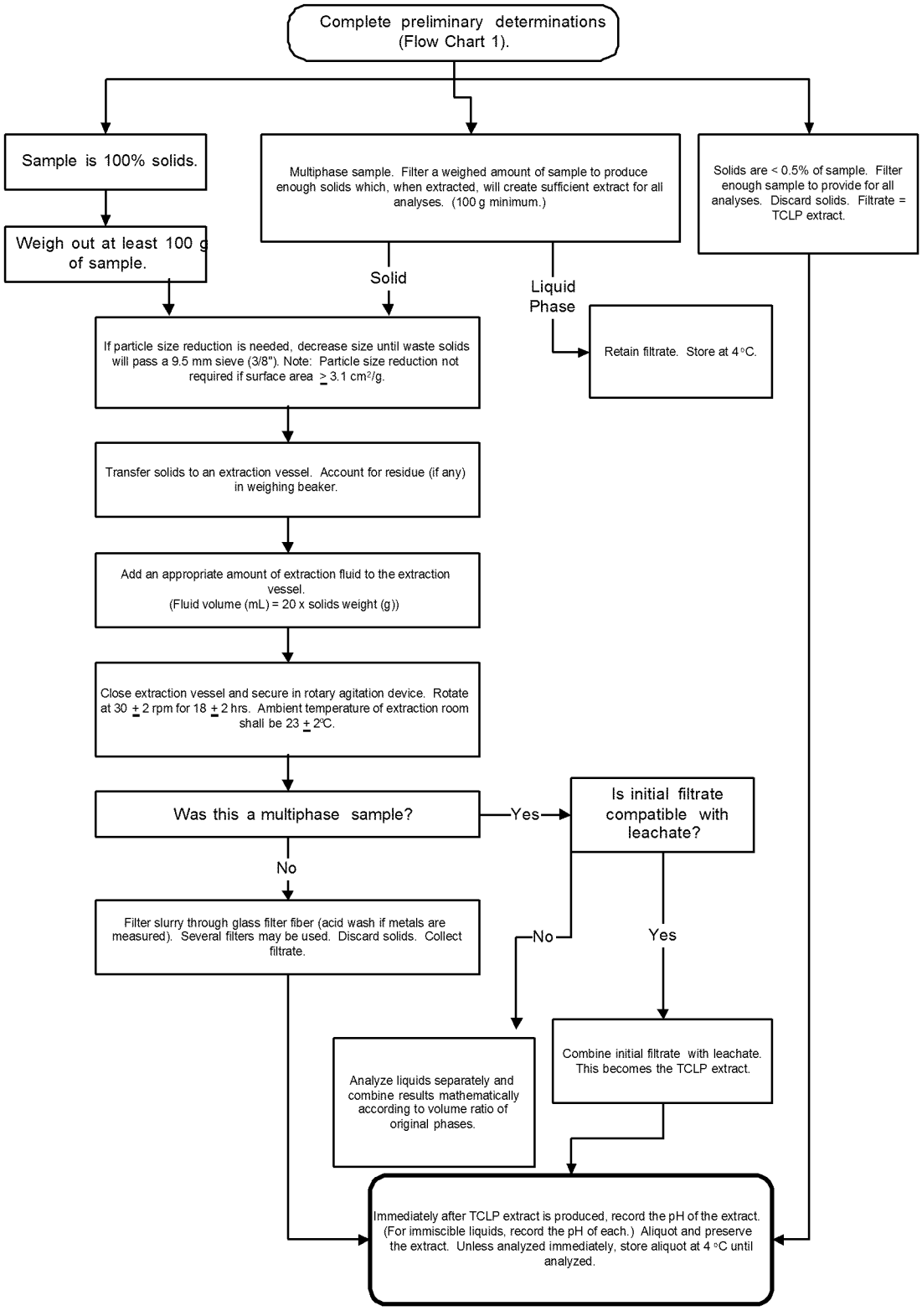
APPENDIX D

FLOW CHARTS

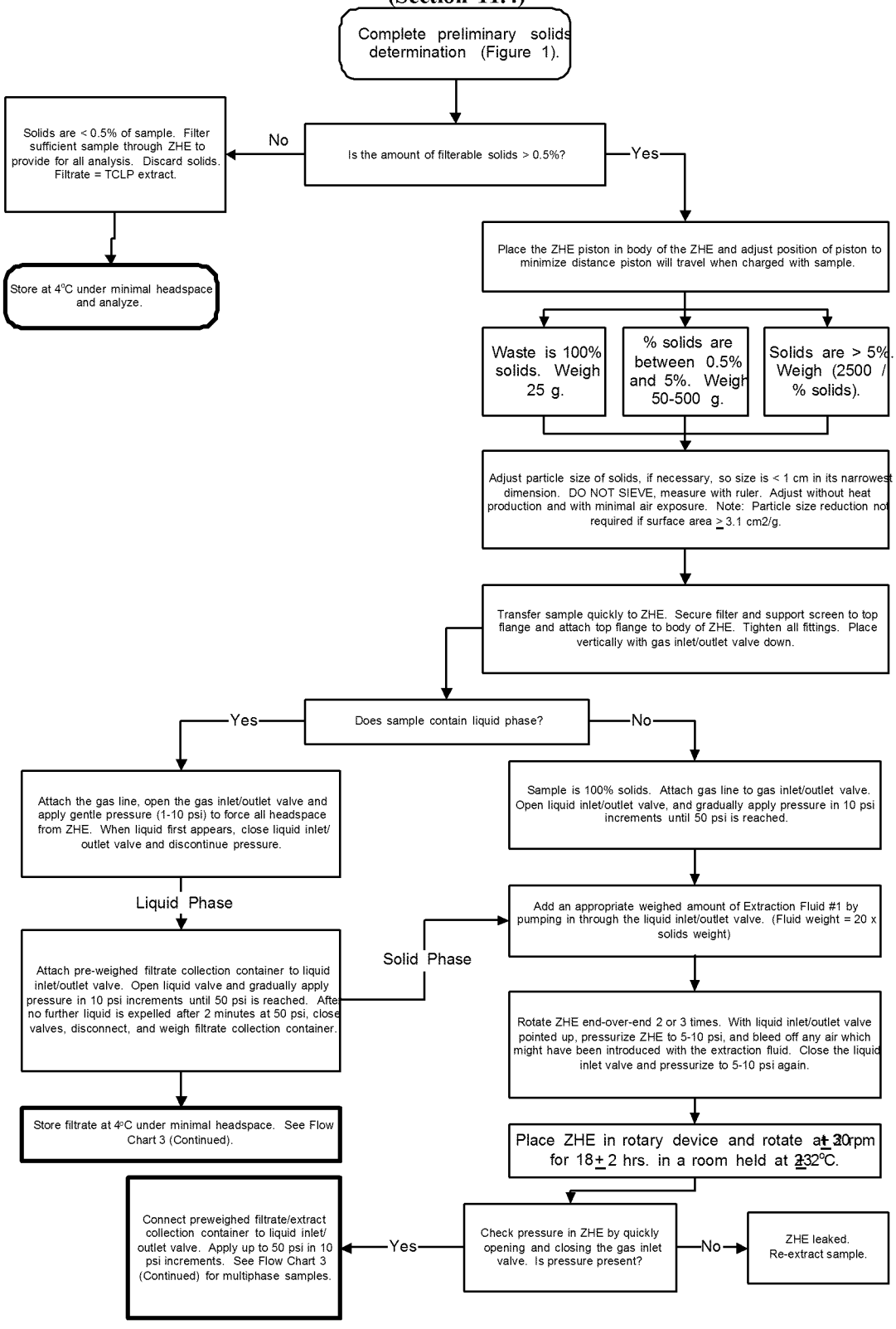
Flow Chart 1. Preliminary Sample Evaluation (Section 11.2)



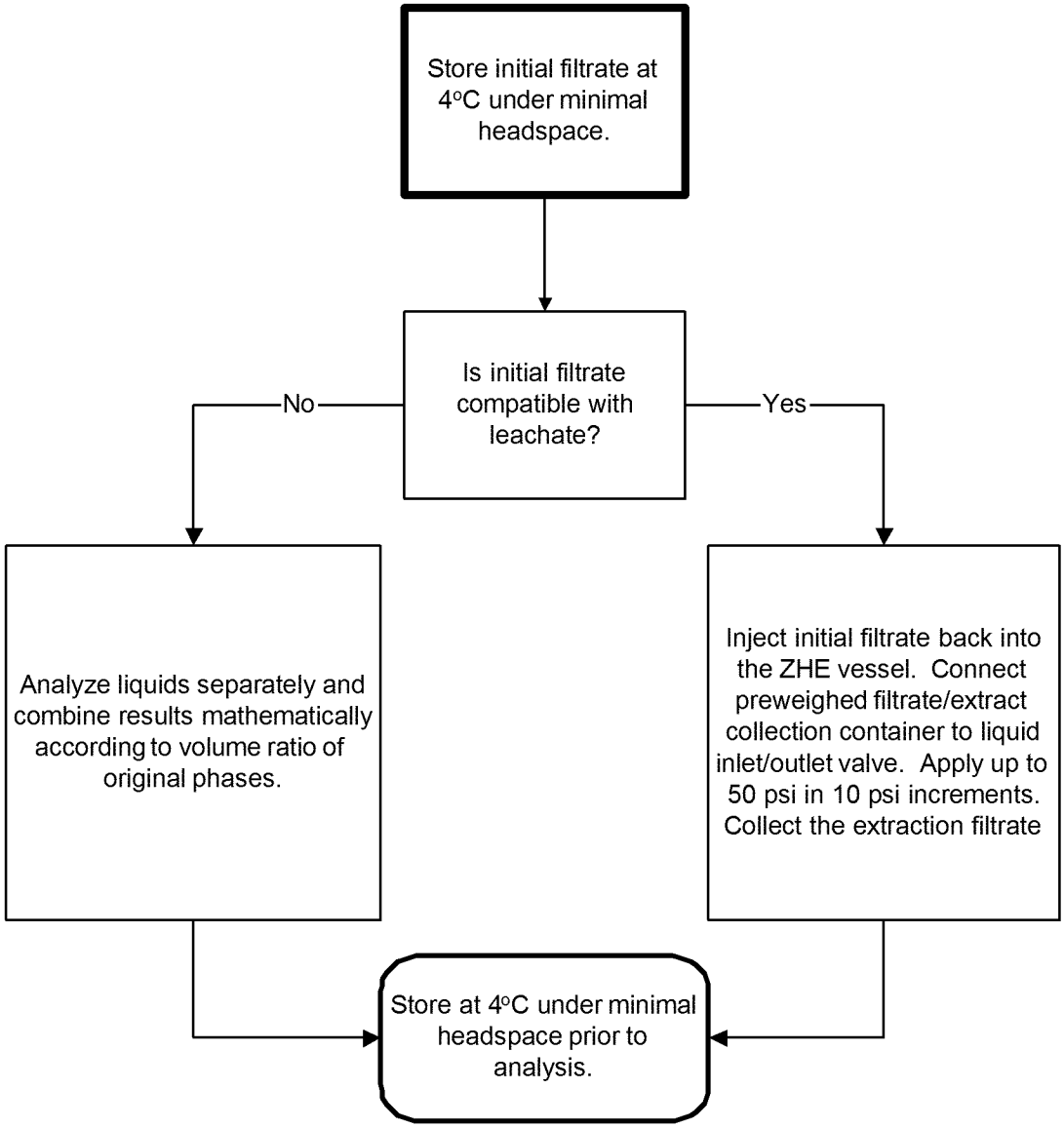
Flow Chart 2. Bottle Extraction, Non-Volatile Constituents (Section 11.3)



Flow Chart 3. ZHE Extraction, Volatile Constituents (Section 11.4)



Flow Chart 3. ZHE Extraction
(Continued)



Appendix F

Laboratory Support Standard Operating Procedures

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Document No. NC-QAM-001, Rev. 2-A
Effective Date: 05/23/12
Cover Page Page 1 of 274

Quality Assurance Manual

Ohio EPA has not yet reviewed this revision for use under the OH VAP program.

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Quality Assurance Manual
Approval Signatures

Laboratory Director – Opal Davis-Johnson	04/23/12 Date
Quality Assurance Manager – Dorothy Leeson	05/23/12 Date
Technical Director – Dr. Mark Bruce	05/22/12 Date

SECTION 2. TABLE OF CONTENTS

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7.0 SERVICE TO THE CLIENT		V1M2 Secs. 4.4.1 - 4.4.4	4.4.1; 4.4.2; 4.4.3; 4.4.4		
7.1	Overview	V1M2 Secs. 4.4.5; 4.5.5; 5.7.1	4.4.5; 5.7.1		
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SOP/Policy Reference	Title
CA-C-S-001	Work Sharing Process
CW-L-P-004	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-L-S-002	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-006	Detection Limits
CA-Q-S-008	Management Systems Review
CA-T-P-001	Qualified Products List
CW-E-M-001	Corporate Environmental Health & Safety Manual
CW-F-P-002	Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CW-F-S-007	Controlled Purchases Policy
CW-F-S-018	Vendor Selection
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CA-Q-M-002	Corporate Quality Management Plan
NC-QA-015	Equipment Monitoring and Thermometer Calibration
NC-QA-018	Statistical Evaluation of Data and Development of Control Charts
NC-QA-019	Records Information Management
NC-QA-027	Preparation and Management of Standard Operating Procedures
NC-QA-028	Employee Orientation and Training
NC-QA-029	Nonconformance and Corrective Action System
NC-SC-005	Sample Receiving and Sample Control
NC-SC-006	Sample Procurement Protocol
CA-Q-T-005	Laboratory Documentation
NC-QA-021	Evaluation of Method Detection Limits for Chemical Tests

SECTION 3

INTRODUCTION, SCOPE, AND APPLICABILITY

3.1 INTRODUCTION AND COMPLIANCE REFERENCES

TestAmerica North Canton's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with the NELAC Institute (TNI) Standard, dated 2009, Volume 1, Modules 2 and 4 and ISO/IEC Guide 17025:2005(E). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan, CA-Q-M-002, (CQMP) and the various accreditation and certification programs listed in Appendix 4. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations. The relevant NELAC section is included in the heading of each QAM section.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.
- U.S. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 4.2, October 2010.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- Toxic Substances Control Act (TSCA).

3.2 TERMS AND DEFINITIONS

A Quality Assurance Program is a company-wide system designed to ensure data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 3 for the Glossary/Acronyms.

3.3 SCOPE / FIELDS OF TESTING

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among effluent water, groundwater, hazardous waste, sludge, wipes, and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Appendix 4. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory must abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director, the Quality Assurance (QA) Manager, and the Technical Director. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

- Specific requirements delineated in project plans may supersede general quality requirements described in this manual. Ohio VAP requirements are listed throughout the document.

3.4 MANAGEMENT OF THE MANUAL

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Corporate Quality Management personnel to assure it remains in compliance with Section 3.1. This manual itself is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager must review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates must be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control and Updating Procedures (refer to SOP NC-QA-027 Preparation and Management of SOPs).

SECTION 4

MANAGEMENT REQUIREMENTS

4.1 OVERVIEW

TestAmerica North Canton is a local operating unit of TestAmerica Laboratories, Inc. The organizational structure, responsibilities, and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President, Chief Operating Officer, Corporate Quality Assurance, Corporate Quality Director, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate and TestAmerica North Canton is presented in Figure 4-1 Employee names are provided to demonstrate range and size of departments however the actual staff members may vary over time. The most current Organization Chart may be obtained from Quality Assurance Manager or Laboratory Director. .

4.2 ROLES AND RESPONSIBILITIES

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program. More extensive job descriptions are maintained by laboratory management.

4.2.1 Additional Requirements for Laboratories

The responsibility for quality resides with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for knowing the content of this manual and upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica North Canton laboratory.

North Canton Laboratory Key Personnel

Name	Position
Rusty Vicinie	General Manager, NE Region
Opal Davis-Johnson	Laboratory Director
Raymond Risden	Operations Manager
Dorothy Leeson	Quality Assurance Manager
Mark Bruce	Technical Director
Rebecca Strait	Client Services Director

Steve Jackson	Regional Safety Director, Waste Management Supervisor
Olguita Colon	General Chemistry Group Leader
Will Cordell	Field Analytical Group Leader
Steve Earle	Extractions Group Leader
Al Haidet	Shipping Group Leader
Dave Heakin	Speciality Analysis Group Leader
Tom Hula	GC/MS Semivolatiles Group Leader
Chris Livengood	Sample Control Group Leader
Darren Miller	Maintenance Group Leader
Susan Girard	Metals Group Leader
Carolynne Roach	GC Volatile and GC Semivolatiles Group Leader
Tom Stiller	GC/MS Volatiles Group Leader
Patrick O'Meara	Project Management Group Leader

4.2.2 Quality Assurance (QA) Manager or Designee

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system.

The QA Manager reports directly to the Laboratory Director, and has access to Corporate QA for advice and resources. This position is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications, and other quality assurance related items. The QA Manager directs the activities of the QA officers to accomplish specific responsibilities, which include, but are not limited to:

- Serves as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory oeprations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications, scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.

- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action database and the corrective and preventive action systems.
-
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12; and if deemed necessary, may be temporarily suspended during the investigation.
- Objectively monitoring standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDL, control limits, and miscellaneous forms and information.
- Review a percentage of all final data reports for internal consistency. Review of Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, 5% of calculations, format, holding time, sensibility and completeness of the project file contents.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Captains the QA team to enable communication and to distribute duties and responsibilities.
- Ensuring communication and monitoring standards of performance to ensure systems are in place to produce the level of quality as defined in this document.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 12.
- Evaluation of the thoroughness and effectiveness of training.
- Compliance with ISO 17025.

4.2.2 Technical Director & Department Group Leader

The Technical Director reports directly to the Laboratory Director. The Technical Director along with the Laboratory Director, the QA Manager, the Operations Manager, and each Deparment

Group Leader is accountable for compliance with the ISO 17025 Standard. The Technical Director works with QA and Department Group Leaders to solve day to day technical issues, provide technical training and guidance to laboratory staff, project managers, and clients, and assists with method development and validation.

The Department Group Leaders report to the Operations Manager. The Group Leaders maintain overall responsibilities for a defined portion of the laboratory. These responsibilities include but are not limited to:

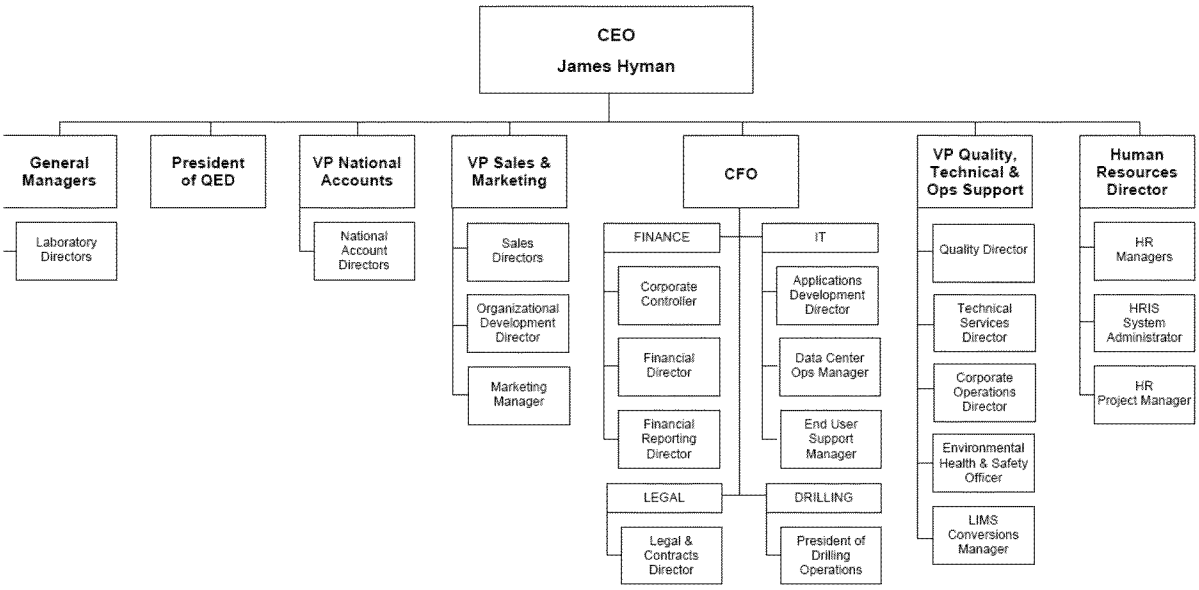
- Day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Working with the QA Manager to coordinate preparation of test method SOPs and performs subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples.
- Monitoring the validity of the analyses performed and data generated in the laboratory.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved LIMS untilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Working with the QA Manager in scheduling all QA/QC-related requirements for compliance., e.g. MDLs, etc.

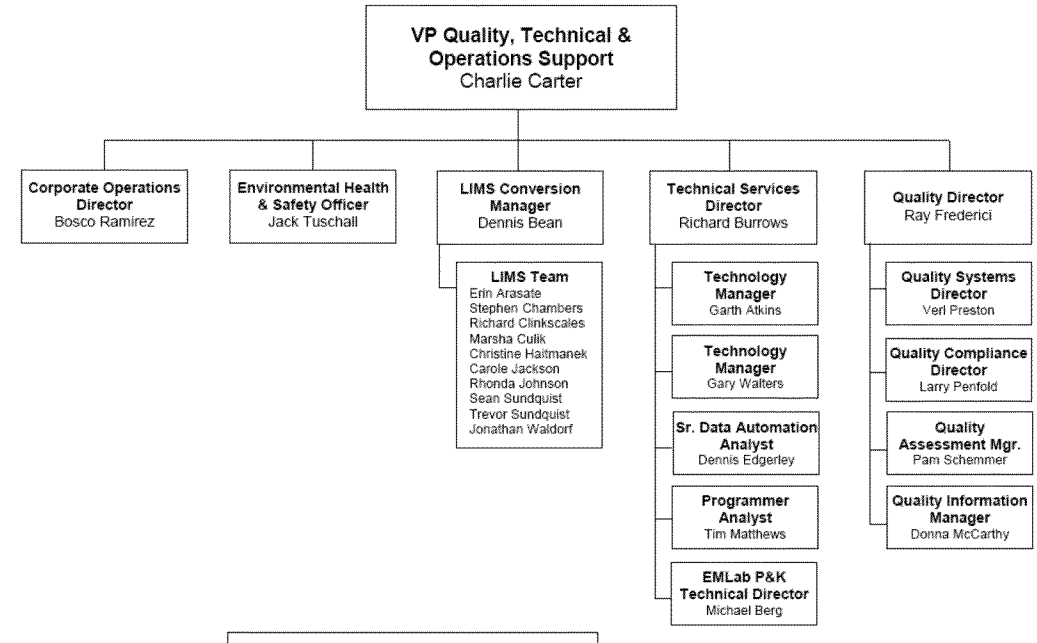
4.3 DEPUTIES

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy
Laboratory Director	Technical Director
Quality Assurance Manager	Quality Assurance Specialist
Technical Director	Quality Assurance Manager
EHS Coordinator	Operations Manager

Figure 4-1. Corporate and Laboratory Organization Charts




VP Quality, Technical & Operations Support
Charlie Carter
Corporate Operations Director
Bosco Ramirez
Environmental Health & Safety Officer
Jack Tuschall
LIMS Conversion Manager
Dennis Bean
LIMS Team
Erin Arasate
Stephen Chambers
Richard Clinkscales
Marsha Culik
Christine Haitmanek
Carole Jackson
Rhonda Johnson
Sean Sundquist
Trevor Sundquist
Jonathan Waldorf
Technical Services Director
Richard Burrows
Technology Manager
Garth Atkins
Technology Manager
Gary Walters
Sr. Data Automation Analyst
Dennis Edgerley
Programmer Analyst
Tim Matthews
EMLab P&K Technical Director
Michael Berg
Quality Director
Ray Frederici
Quality Systems Director
Verl Preston
Quality Compliance Director
Larry Penfold
Quality Assessment Mgr.
Pam Schemmer
Quality Information Manager
Donna McCarthy
Note:
QA Managers and EH&S Managers have a direct reporting relationship to both operations leadership and corporate functional leadership.
4 Aug 2011

SECTION 5

QUALITY SYSTEM

5.1 QUALITY POLICY STATEMENT

It is TestAmerica's policy to:

Provide data of known qualty to its clients by adhering to approved methodologies, regulatory requirements, and the QA/QC protocols.

Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.

Continually improve systems and provide support to quality improvement efforts in laboratory, administrative, and managerial activities. TestAmerica recognizes that the implementation of a Quality Assurance program requires management's commitment and support as well as the involvement of the entire staff.

Provide clients with the highest level of professionalism and the best service practices in the industry.

To comply with the ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard, and to continually improve the effectiveness of the management system.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 ETHICS AND DATA INTEGRITY

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of the TestAmerica Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy CW-L-P-004) and Employee Ethics Statements (Appendix 1)
- Ethics and Compliance Officers (ECOs)
- A training program
- Self-governance through disciplinary action for violations
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct (Corporate SOPCW-L-S-002)
- Procedures and guidance for recalling data if necessary (Corporate SOPCW-L-S-002)
- Effective external and internal monitoring system that includes procedures for internal audits (Section 16)

- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 QUALITY SYSTEM SUPPORTING DOCUMENTATION

The laboratory's Quality System is communicated through a variety of documents

- Quality Assurance Manual – Each laboratory has a lab-specific Quality Assurance Manual.
- Corporate SOPs and Policies - Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions - A subset of procedural steps, tasks or forms associated with an operation of a management system, e.g., checklists, preformatted bench sheets, forms.
- Laboratory SOPs – General and technical
-
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

-
- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other: Work Instructions (WI), memos, flow charts, etc.

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's (QAM) shall take precedence over the CQMP in those cases. Any regulatory requirements (e.g.; Ohio VAP, CT RCP, etc) provided in the laboratory specific documents (i.e., QAM and SOPs) take precedence over any policies provided in corporate documents.

5.4 QA/QC OBJECTIVES FOR THE MEASUREMENT OF DATA

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory must provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS). Equations to derive relevant QC objectives can be found in the method specific SOPs.

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS.

A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project) expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability must be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), inter-element corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc.

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

5.5 CRITERIA FOR QUALITY INDICATORS

The laboratory maintains Quality Control Limits in LIMS. Reports t that summarizes the precision and accuracy acceptability limits for performed analyses can be generated from LIMS. This summary includes an activation date, is updated each time new limits are generated, and are managed by the laboratory's QA Department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where U.S. EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in NC-QA-018 Statistical Evaluation of Data and Development of Control Charts and in Section 24).

5.6 STATISTICAL QUALITY CONTROL

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Group Leader and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance Department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 25. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

In-house limits for all QC data must be evaluated and compared to the limits published in the methods for applicable matrices. Method limits must be employed until sufficient QC data are

acquired. A minimum of 20 to 30 data points are recommended to establish the in-house QC limits. Calculated results of the QC (LCS) samples are evaluated by comparing against control limits (3-sigma).

Control charts are used to develop control limits, trouble-shoot analytical problems, and, in conjunction with the non-conformance system, to monitor for trends. Program-specific data analysis requirements for control charts are followed as required for data generated under those programs. These additional requirements shall be documented in a QAPP.

5.7 QUALITY SYSTEM METRICS

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6

DOCUMENT CONTROL

6.1 OVERVIEW

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled at each laboratory Facility:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers, and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the company intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP NC-QA-027, Preparation and Management of Standard Operataing Procedures.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. The laboratory also maintains instrument manuals (hard or electronic copies).

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and Corrective Action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item the effective date, revision number, and the laboratory name. The QA Department is responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department and other management. In order to develop a new document, a staff member submits a draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version

information to the document, and retains that docume nt as the official document on file. That document is then provided to all applicable operational units (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures must be reviewed at a minimum of every 24 months, and revised as appropriate. For procedures associated with DoD and Ohio VAP project work, applicable SOPs and Policies are reviewed every 12 months. Changes to documents occur when a procedural change warrants.

6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY

For changes to the QA Manual, refer to SOPs NC-QA-019 and CW-Q-S-001. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA/QC Department. Electronic copies are stored on the Public server in the QAQC folder for the applicable revision.

For changes to SOPs, refer to Corporate SOP CW-Q-S-002, Writing a Standard Operating Procedure (SOP), and SOP NC-QA-027, Preparation and Management of Standard Operating Procedures. The SOP identified above also defines the process of changes to SOPs.

Forms, worksheets, Work Instructions, and information are organized by department in the QA office. Electronic versions are kept on a hard drive in the QA department; hard copies are kept in QA files. The procedure for the care of these documents is in SOP NC-QA-027.

6.4 OBSOLETE DOCUMENTS

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP NC-QA-027.

SECTION 7

SERVICE TO THE CLIENT

7.1 OVERVIEW

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (Percent Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time must be checked for feasibility.

Electronic or hard-copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this must be documented and discussed with the client prior to contract approval (refer to Section 8 for Subcontracting Procedures).

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (S APs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 REVIEW SEQUENCE AND KEY PERSONNEL

Appropriate personnel must review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the opportunity is forwarded to a Customer Service Manager (CSM) for review. The CSM contacts the appropriate Sales Executive (National Account Manager, Key Account Executive, Regional Account Executive, and/or Program Manager) to determine which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, reporting specifications, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP CA-L-P-002, Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Legal & Contracts Director
- Laboratory Customer Service Manager
- Laboratory Operations Manager
- Laboratory and/or Corporate Technical Director
- Laboratory and/or Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality Assurance Managers
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote, and makes final acceptance for their facility.
- Based on the level of discount extended for the project, approval of the General Manager or Sales Director may also be required.

The Customer Service Manager or local Account Executive then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her backup will fulfill the review requirements.

The Legal & Contracts Director (or their designee) maintains copies of all signed contracts. The Laboratory Director also maintains an electronic copy of any contract signed at the local level.

7.3 DOCUMENTATION

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. Documents are reviewed by the Laboratory Director and stored on the laboratory's public drive.

The contract must be distributed to and maintained by the Corporate Contracts Department and the applicable Account Executive. A copy of the contract must be filed electronically by the Laboratory Director. Quotes must be archived electronically in the laboratory quote module (TALs) or in the public shared drive if an off-TALs quote is submitted.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps email records or a phone log of conversations with the client.

7.3.1 Project-Specific Quality Planning

Communication of contract-specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes, e.g., use of a non-standard method or modification of a method, and approvals must be documented prior to implementation.

Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory. Project-specific changes made after samples are in-house are communicated through Change Information Notification emails

Programmatic and/or method changes are communicated via email transmittal and/or in meetings with the applicable Operations Managers. If the modification includes use of a non-standard method, or significant modification of a method, documentation of the modification is made in the case narrative of the applicable data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 SPECIAL SERVICES

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

Note: ISO/IEC 17025:2005(E) states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third-party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5 CLIENT COMMUNICATION

Customer Service Managers (CSMs) and Project Managers (PMs) are the primary communication link to the clients. They must inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project Management must maintain ongoing client communication throughout the entire client project.

Technical Directors, Operation Manager, or Group Leaders are available to discuss any technical questions or concerns the client may have.

7.6 REPORTING

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 CLIENT SURVEYS

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

TestAmerica Sales and Marketing teams periodically develop lab and client-specific surveys to assess client satisfaction.

SECTION 8

SUBCONTRACTING OF TESTS

8.1 OVERVIEW

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to theTestAmerica Laboratories. The phrase "work sharing" refers to internal transfers of samples between the TestAmerica Laboratories. The term "outsourcing" refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances , we must be assured that the subcontractors or work sharing laboratories understand the requirements and must meet the same commitments we have made to the client. Refer to TestAmerica's Corporate SOPs on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process SOP (CA-C-S-001).

When outsourcing analytical services, the laboratory must assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI ISO/IEC 17025:2005(E) and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation must be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work must be identified in the final report, as must non-TNI accredited work, where required.

For DOD projects, the subcontractor laboratories used must have an established and documented laboratory quality system that complies with DoD QSM requirements. The subcontractor laboratories are evaluated following the procedures outlined below and as seen in Figure 8-2. The subcontractor laboratory must receive project-specific approval from the DoD client before any samples are analyzed.

The QSM has five specific requirements for subcontracting:

1. Subcontractor laboratories must have an established laboratory quality system that complies with the QSM.

2. Subcontractor laboratories must be approved by the specific DoD component laboratory approval process.

3. Subcontractor laboratories must demonstrate the ability to generate acceptable results from the analysis of PT samples, subject to availability, using each applicable method, in the specified matrix, and provide appropriate documentation to the DoD client.

4. Subcontractor laboratories must receive project-s pecific approval from the DoD client before any samples are analyzed.

5. Subcontractor laboratories are subject to project-specific, on-site assessments by the DoD client or their designated representatives.

Project Managers (PMs) or Customer Service Managers (CSM) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory must advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client must be retained in the project folder.

Note: In addition to the client, some regulating agencies (e.g., USDA) or contracts (e.g., certain USACE projects) may require notification prior to placing such work.

8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

Whenever a PM or Customer Service Manager (CSM) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory
- Firms specified by the client for the task. (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder.)
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica. A listing of all approved subcontracting laboratories is available on the TestAmerica intranet site. Supporting documentation is maintained by Corporate offices and by the Testamerica laboratory originally requesting approval of the subcontract lab. Verify necessary accreditation, where applicable (e.g., on the subcontractors TNI , A2LA accreditation, or State Certification.
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses
- TNI or A2LA-accredited laboratories
- In addition, the firm must hold the appropriate certification to perform the work required

All TestAmerica Laboratories are pre-qualified for work sharing, provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. Refer to Corporate SOP CA-C-S-001, Work Sharing Process.

When the potential subcontract laboratory has not been previously approved, CSMs or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that

the QA Manager begin the process of approving the su bcontract laboratory as outlined in Corporate SOP CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility. (An e-mail is sufficient documentation; or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented.)

8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to Corporate Contracts for formal contracting with the laboratory. They must add the lab to the approved list on the intranet site, and notify the Finance Group for J.D.Edwards.

8.2.2 The client must assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list, and can only be recommended to the extent that we would use them.

8.2.3 The status and performance of qualified subcontractors must be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified must be brought tothe attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints must be investigated. Documentation of the complaint, investigation, and corrective action must be maintained in the subcontractor file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information must be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing must be retained on the intranet listing. The QA Manager must notify all TestAmerica laboratories, Corporate Quality, and Corporate Contracts if any laboratory requires removal from the intranet site. This notification must be posted on the intranet site and e-mailed to all Laboratory Directors, QA Managers, and Sales Personnel.

8.3 OVERSIGHT AND REPORTING

The CSM or PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM or CSM responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented on a Subcontracted Sample Form (Figure 8-1), and the form is retained in the project folder. For

TestAmerica Laboratories, certifications can be view ed on the company's TotalAccess Database.

The Sample Control Department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must also be included with all samples workshared within TestAmerica. Client COCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratory EDD, i.e., imported, the report must explicitly indicate the specific lab that produced the data and identify the specific methods and samples.

Note: The results submitted by a TestAmerica work-sharing laboratory may be transferred electronically and the results reported by the TestAmerica work-sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 CONTINGENCY PLANNING

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs; however, this decision and justification must be documented in the project files, and the "Purchase Order Terms and Conditions for Subcontracted Laboratory Services" must be sent with the samples and Chain-of-Custody. In the event this provision is utilized, the laboratory (e.g., QA Manager) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

Figure 8-1. Example - Client-Approved Subcontractor Form

Client Information:

Client Name & Account Number:_____

Client Contact: _____

Client Address: _____

_____

Project Information: **(Please choose all applicable.)**

Certification required: ☐ **State** ☐ **TNI** ☐ **A2LA** ☐ **Method**___

☐ **Target compound**_____ ☐ ☐ **Other**_____ ☐ **N/A**

Required Turn around time (method provisional)_____

Subcontractor's Information:

Subcontractor's Name: _____

Subcontractor's Contact: _____

Subcontractor's Email: _____

Subcontractor's Address: _____

_____

Subcontractor's Phone Number: _____

Analytical Test/Compound/Method to be subcontracted: _____

Certification Statement:

I hereby give ***[Insert Lab Name]*** permission to use the above noted subcontractor for the above noted testing procedures/methods. I realize that the above subcontractor will be held liable for the validity of the above mentioned testing procedures/methods. All subcontractors shall meet the requirements as spelled out in project information and will follow all analytical holding times and turn around times for analytical reports. The subcontract laboratory, and not TestAmerica, will be held liable for liquidated damages for delays in subcontracted analytical reports and/or electronic data deliverables.

Client Signature **Date**

SECTION 9

PURCHASING SERVICES AND SUPPLIES

9.1 OVERVIEW

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with the TestAmerica's Corporate Controlled Purchases Procedure, SOP CW-F-S-007.

Contracts must be signed in accordance with TestAmerica's Corporate Authorization Matrix Policy, Policy CW-F-P-002. Request for Proposals (RFP's) must be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 GLASSWARE

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass must be used where possible. For safety purposes, thick-wall glassware must be used where available.

9.3 REAGENTS, STANDARDS & SUPPLIES

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent and Acid Lot Testing and Approval, SOP CA-Q-S-001.

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. The analyst may check the item out of the on-site consignment system that contains items approved for laboratory use. If

the item is not in consignment, the analyst must pro vide the master item number, item description, package size, catalogue page number, and the quantity needed. If an item being ordered is not the exact item requested, approval must be obtained from the Operations Manager or Group Leader prior to placing the order. The purchasing manager places the order.

9.3.2 Receiving

It is the responsibility of the Warehouse Manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials where received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Material Safety Data Sheets (MSDSs) are kept on a backup disc located in the Wet Chemistry bullpen and available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

Methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent. Specifications are listed in SOP NC-QA-017, Reagents and Standards.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five-year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents must not be used past the manufacturer's or SOP's expiration date unless "verified" (refer to Item 3 listed below).

- An expiration date cannot be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.

- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Method Blanks, Laboratory Control Sample (LCS), etc.).

- If the dry chemical/solvent is used for the preparation of standards, the expiration dates can be extended six months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison studies are maintained in the Reagent module of LIMS for each laboratory group.

Wherever possible, standards must be traceable to na tional or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. The minimum total pressure must be 500 psig, or the tank must be replaced. To prevent a tank from going to dryness, close observation of the tank gauge must take place as pressure decreases towards 500 psig, or the tank myust be replaced. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a conductivity of less than 1 μmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25 °C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Operations Manager and appropriate Technical Manager must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased bottleware used for sampling must be certified clean, and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage condictions are per the Corporate Environmental Health & Safety Manual (Corporate Document CW-E-M-001) and method SOPs or manufacturer instructions.

9.4 PURCHASE OF EQUIPMENT/INSTRUMENTS/SOFTWARE

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or group leader makes a supply request to the Operations Manager and/or the Laboratory Director. If they agree with the request the procedures outlined in TestAmerica's Corporate Policy CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed, and Purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned, such as HP-20, and added to the equipment list described in Section 21 that is maintained by the QA Department, and I.T. must be notified so they can synchronize the instrument for backups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated followed by MDLs, Demonstration of Capabilities

(DOCs), and other relevant criteria (refer to Sectio n 20). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

9.5 SERVICES

Service to analytical instruments (except analytical balances) is performed on an as-needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Department Managers. The service providers that perform the services are approved by the Department Managers or Operations Manager.

9.6 SUPPLIERS

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP CW-F-S-018) and Procurement and Contracts Policy (Policy CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers /vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report (CW-F-WI-009).

The Corporate Purchasing Group must work through the appropriate channels to gather the information required to clearly identify the problem and must contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports must be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Laboratory Director are consulted with vendor and product selection that have an impact on quality.

SECTION 10

COMPLAINTS

10.1 OVERVIEW

The laboratory considers an effective client complaint handling process to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and improving client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services, (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following SOPs NC-QA-029, Nonconformance and Corrective Action System, and CA-C-S-002, Complaint Handling and Service Recovery.

10.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to CA-C-S-002, Complaint Handling and Service Recovery.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints must be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory must inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.3 INTERNAL COMPLAINTS

Internal complaints include, but are not limited to errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and must follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing, and Information Technology (IT) may initiate a complaint by contacting the laboratory or through the Corrective Action system described in Section 12.

10.4 MANAGEMENT REVIEW

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16)

SECTION 11

CONTROL OF NON-CONFORMING WORK

11.1 OVERVIEW

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies, and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a Corrective Action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the Corrective Action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's Corrective Action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it with the Technical Director or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Operations Manager and QA Manager, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with TNI (or the analytical method) requirements and the reason. Data being reported to a non- TNI state would need to note the change made to how the method is normally run.

Note: The laboratory must implement Corrective Action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all Corrective Actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale will be presented in the final report and/or affidavit and the data will be noted as 'not VAP certified' on the affidavit.

11.2 RESPONSIBILITIES AND AUTHORITIES

TestAmerica's Corporate SOP entitled Internal Investigation of Potential Data Discrepancies and Determination for Data Recall (SOP CW-L-S-002) outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or

violations of the TestAmerica's data integrity polic ies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances the Laboratory Director, Operations Manager, Project Manager, or a member of the QA team may exceptionally authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client must be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's Corrective Action procedures described in Section 13. This information may also need to be documented in logbooks and/or data review as appropriate . Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24 hours. The Senior Management staff is compromised of the Laboratory Director, QA Manager, Customer Service Manager, Operations Manager, I.T. Manager, H.R. Manager, PM Manager, and Technical Director. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), Director of Quality and Client Advocacy, and the laboratory's Corporate Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, Corporate Quality Director, the COO, General Managers, and the Corporate Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation and Recall Procedure (SOPCW-L-S-002) distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECOs and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/Corrective Action reporting in lieu of the data recall determination form contained in TestAmerica Corporate SOPCW-L-S-002.

11.4 PREVENTION OF NONCONFORMING WORK

If it is determined that the nonconforming work coul d recur, further corrective actions must be made following the laboratory's Corrective Action system. Periodically, as defined by the laboratory's preventive action schedule (monthly), the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's Corrective Action process may be followed.

11.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)

In some cases it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality must be respected, and the problem with the required corrective and preventive action must be stated in writing and presented to the Laboratory Director.

The Laboratory Director must arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting must be held to confirm that there is a problem, that suspension/restriction of the method is required and must be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target, or test fully back on line.

The QA Manager must also initiate a Corrective Action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed-upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the Internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction, i.e., Project Management, Log-in, etc. Clients must NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager must determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Director, QA Manager, Group Leader) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed Corrective Action report.

SECTION 12

CORRECTIVE ACTION

12.1 OVERVIEW

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the Corrective Action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Nonconformance Memos (NCM).

12.2 GENERAL

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a Corrective Action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution

12.2.1 Non-Conformance Memo (NCM)

An NCM is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Client Complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips

12.2.2 Corrective Action Report (CAR) – can be used to document the following types of corrective actions:

- Questionable trends that are found in the review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors

- Client complaints
- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports

This will provide background documentation to enable root cause analysis and preventive action.

12.3 CLOSED LOOP CORRECTIVE ACTION PROCESS

Any employee in the company can initiate a Corrective Action. There are four main components to a closed-loop Corrective Action process once an issue has been identified--Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

Upon discovery of a non-conformance event, the event must be defined and documented. An NCM must be initiated, someone is assigned to investigate the issue, and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment. SOP NC-QA-029, Nonconformance and Corrective Action System, establishes procedures for the identification and documentation of nonconformances and the corrective actions taken as a result of these events.

The cause analysis step is the key to the process as a long-term corrective action cannot be determined until the cause is determined.

If the cause is not readily obvious, the Group Leader, Technical Director, Lab Director, QA Manager, or designee is consulted.

12.3.2 Selection and Implementation of Corrective Actions

Where corrective action is needed, the laboratory must identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.

Corrective actions must be, to a degree, appropriate to the magnitude of the problem identified through the cause analysis.

Whatever corrective action is determined to be appropriate, the laboratory must document and implement the changes. The NCM is used for this documentation.

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a

significant failure. The root cause may be buried un der seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness.

Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the Root Cause data from these incidents to identify root causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred five consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

- Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

12.3.4 Monitoring of the Corrective Actions

- The Group Leader or Technical Director and QA Manager is responsible to ensure the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. The Technical Director are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM is entered into a database for tracking purposes and a summary of all corrective actions is reviewed to aid in ensuring the corrective actions have taken effect.
- The QA Manager reviews monthly NCMs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and must be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

Also refer to Section 15.2.4, Special Audits.)

12.4 TECHNICAL CORRECTIVE ACTIONS

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11 for information regarding the control of non-conforming work). The documentation of these procedures is through the use of an NCM.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, and QAM Sections 19 and 20. The QA Manager reviews all corrective actions monthly, at a minimum, and highlights are included in the QA monthly report.

To the extent possible, samples must be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data must be reported with an appropriate data qualifier and/or the deficiency must be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by a written NCM and appropriate corrective action (e.g., re-analysis) is taken and documented

12.5 BASIC CORRECTIONS

When mistakes occur in records, each mistake must be crossed-out, and [not obliterated (e.g. no White-Out)], and the correct value entered alongside. All such corrections must be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original “uncorrected” file must be maintained intact and a second “corrected” file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) must also be documented.

Table 12-1 .
General Corrective Action Procedures

*** For the Ohio EPA Voluntary Action Program (VAP), please refer to the SOPs for the acceptable criteria, corrective actions, and exceptions.**

INORGANIC LABORATORY QUALITY CONTROL SAMPLES

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]
Alkalinity	* Method Blank	310.1 2320B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	—	N/A
	Laboratory Control Sample	310.1 2320B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	310.1 2320B	Total alkalinity: 1 per batch of 20 samples	—	N/A
	Matrix Spike Duplicate	310.1 2320B	Total alkalinity: 1 per batch of 20 samples	—	N/A
	Duplicate	310.1 2320B	For carbonate, bicarbonate, hydroxide, alkalinity, and total alkalinity by SM2320B . Frequency: 1 per batch of 10 samples Criteria 310.1: ≤ 20 % RPD(3) Criteria 2320B: ≤ 25 % RPD(3) Corrective Action: Flag data outside of limit.	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]
Acidity	* Method Blank	305.1 SM2310 B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank.	—	N/A
	Laboratory Control Sample	305.1 SM2310 B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	305.1 SM2310 B	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Matrix Spike Duplicate	305.1 SM2310 B	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	305.1 SM2310 B	N/A	—	N/A

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Ammonia *	Method Blank	350.2 350.3 SM4500 NH-E,F	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	—	N/A
	Laboratory Control Sample	350.2 350.3 SM4500 NH-E,F	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	—	N/A
	Matrix Spike	350.2 350.3 SM4500 NH-E,F	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Ammonia (Cont'd)	Matrix Spike Duplicate	350.2 350.3 SM4500 NH-E,F	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	350.2 350.3 SM4500 NH-E,F	N/A	—	N/A
Ammonia (TKN)	* Method Blank	351.3 SM4500 N-Org C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Ammonia (TKN) (Cont'd)	Laboratory Control Sample	351.3 SM4500 N Org C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	351.3 SM4500 N Prg C	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Matrix Spike Duplicate	351.3 SM4500 N Org C	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	351.3 SM4500 N Org C	N/A	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]
BOD	* Method Blank	405.1 SM5210B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	—	N/A
	Laboratory Control Sample	405.1 SM5210B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	405.1 SM5210B	N/A	—	N/A
	Matrix Spike Duplicate	405.1 SM5210B	N/A	—	N/A
	Duplicate	405.1 SM5210B	N/A	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Anions – Bromide, Chloride, Fluoride, Sulfate, Nitrate, Nitrite, o-Phosphate	* Method Blank	300.0 [(4)]	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank
	Laboratory Control Sample	300.0 [(4)]	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples
	Matrix Spike	300.0 [(4)]	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with MS outside of limit
	Matrix Spike Duplicate	300.0 [(4)]	N/A	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with MS outside of limit

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]	
	Duplicate	300.0 [4]	N/A	9056A	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]	
Arsenic Speciation	* Method Blank			7063	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank.
	Laboratory Control Sample			7063	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike			7063	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit
	Matrix Spike Duplicate			7063	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]
Chemical Oxygen Demand (COD)	Duplicate	410.4 SM5220D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank.	7063	N/A
	* Method Blank			—	N/A
	Laboratory Control Sample	410.4 SM5220D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	410.4 SM5220D	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]
	Duplicate	410.4 SM5220D	N/A	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Chloride *	Method Blank	325.2 SM 4500 Cl E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	9251	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank
	Laboratory Control Sample	325.2 SM 4500 Cl E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within control limits, rerun all associated samples	9251	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	SM 4500 Cl E	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9251	Frequency: 1 with each every 10 samples processed. Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with MS outside of limits

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Chloride (cont'd)	Matrix Spike Duplicate	325.2	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9251	\
	Duplicate	325.2 SM 4500 Cl E	N/A	9251	N/A
Chlorine, Residual	* Method Blank	330.5 SM 4500 Cl G	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	—	N/A
	Laboratory Control Sample	330.5 SM 4500 Cl G	N/A	—	N/A
	Matrix Spike	330.5 SM 4500 Cl G	N/A	—	N/A
	Matrix Spike Duplicate	330.5 SM 4500 Cl G	N/A	—	N/A
	Duplicate	330.5 SM 4500 Cl G	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD(3) Corrective Action: Flag data outside of limit.	—	Water

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]
Chromium (Cr^{+6})	* Method Blank	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank unless the method blank is above RL, and samples are ND.	7196A 3060A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank unless the method blank is above RL, and samples are ND.
	Laboratory Control Sample	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	7196A 3060A	Frequency: 1 with each batch of samples processed not to exceed 20 samples prepped Criteria: percent recovery for water must be within ± 15 % and for solids must be within ± 20% Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	3060A 7196A	Frequency: 1 with each batch of water samples processed not to exceed 20 samples Criteria: Advisory limits are 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike The Method of Standard Addition is used for solid samples in lieu of a Matrix Spike.

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]
Chromium (Cr^{+6}) (Cont'd)	Matrix Spike Duplicate	3500 Cr-D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit	7196A 3060A	Frequency: 1 with each batch of water samples processed not to exceed 20 samples Criteria: Advisory limits are 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike The Method of Standard Addition is used for solid samples in lieu of a Matrix Spike Duplicate.
	Duplicate	3500 Cr-D	N/A	7196A 3060A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: ≤ 20 % RPD(3) limit Corrective Action: Flag data outside of limit.

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]
Conductivity, Specific	* Method Blank	120.1 SM2510B	N/A	9050A Not Applicable
	Laboratory Control Sample	120.1 SM2510B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9050A Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	120.1 SM2510B	N/A	9050A N/A
	Matrix Spike Duplicate	120.1 SM2510B	N/A	9050A N/A
	Duplicate	120.1 SM2510B	Frequency: 1 with each batch of samples processed not to exceed 10 samples Criteria: ≤ 20 % RPD(3) Corrective Action: Flag data outside of limit.	9050A Frequency: 1 with each batch of samples processed not to exceed 10 samples

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Cyanide (Weak Acid Dissociable)	* Method Blank	SM 4500CN-I	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank		
	Laboratory Control Sample	SM 4500CN-I	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples		
	Matrix Spike	SM 4500CN-I	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit		

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
	Matrix Spike Duplicate	SM 4500CN-I	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit		
	Duplicate	SM 4500CN-I	N/A		
Cyanide (Amenable)	* Method Blank	335.1 SM4500C N-G	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	335.1 SM4500C N-G	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	335.1 SM4500C N-G	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9012A	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
	Matrix Spike Duplicate	335.1 SM4500C N-G	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within lab control limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	335.1 SM4500C N-G	N/A	9012A	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]
Cyanide (Total)	* Method Blank	335.2 335.4 4500-CN E 335.2-CLP-M (Ohio VAP)	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank unless the method blank is above RL, and samples are ND.	9012A Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank unless the method blank is above RL, and samples are ND.
	Laboratory Control Sample	335.2 335.4 4500-CN E 335.2-CLP-M (Ohio VAP)	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9012A Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	335.2 335.4 4500-CN E 335.2-CLP-M (Ohio VAP)	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9012A Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]
Cyanide (Total) (cont'd)	Matrix Spike Duplicate	335.2 335.4 4500-CN E 335.2-CLP-M (Ohio VAP)	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9012A Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	335.2 335.4 335.2-CLP-M (Ohio VAP)	N/A	9012A N/A
Dissolved Oxygen	* Method Blank	360.1 SM4500O-G	N/A	— N/A
	Laboratory Control Sample	360.1 SM4500O-G	N/A	— N/A
	Matrix Spike	360.1 SM4500O-G	N/A	— N/A
	Matrix Spike Duplicate	360.1 SM4500O-G	N/A	— N/A
	Duplicate	360.1 SM4500O-G	N/A	— N/A
Flashpoint	* Method Blank	—	N/A	1010 N/A
	Laboratory Control Sample	—	N/A	1010 N/A
	Matrix Spike	—	N/A	1010 N/A

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]	
	Matrix Spike Duplicate	—	N/A	1010	N/A
	Duplicate	—	Frequency: 1 per batch of ≤20 samples per matrix Criteria: RPD[(3)] must be ≤ 20% Corrective Action: Flag data associated with unacceptable Duplicate	1010	Frequency: 1 per batch of ≤20 samples per matrix Criteria: RPD[(3)] must be ≤ 20% Corrective Action: Flag data associated with unacceptable Duplicate

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Fluoride	* Method Blank	340.2 SM4500F-C,ISE	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank		
	Laboratory Control Sample	340.2 SM4500F-C,ISE	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples		

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Flouride (Cont'd)	Matrix Spike	340.2 SM4500F-C,ISE	Frequency: 1 per 10 samples by IC Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit		
	Matrix Spike Duplicate	340.2 SM4500F-C,ISE	Frequency: 1 per 20 samples by IC Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit		
	Duplicate	340.2 SM4500F-C,ISE	N/A		

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Fraction of Organic Carbon	* Method Blank	ASTM D_2974	N/A		
	Laboratory Control Sample	ASTM D_2974	N/A		
	Matrix Spike	ASTM D_2974	N/A		
	Matrix Spike Duplicate	ASTM D_2974	N/A		
	Duplicate	ASTM D_2974	Frequency: 10%		
Hardness	* Method Blank	130.2 SM2340B SM 2340C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	—	N/A
	Laboratory Control Sample	130.2 SM2340B SM 2340C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A

Analysis QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]
Matrix Spike	130.2 SM2340B SM2340C	Method 130.2: 1 per 20 samples Method 2340B: Frequency , Criteria, and Corrective Action See ICP Metals Method 200.7 Requirements	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]
Hardness (cont'd)	Matrix Spike Duplicate	130.2 SM2340B SM 2340C	Method 130.2: 1 per 20 samples Method 2340B: Frequency, Criteria, and Corrective Action: See ICP Metals Method 200.7 Requirements	—	N/A
	Duplicate	130.2 SM2340B SM 2340C	Frequency: One with every 10 samples.	—	N/A
Iron, Ferrous & Ferric	* Method Blank	SM3500-Fe D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	—	N/A
	Laboratory Control Sample	SM3500-Fe D	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Iron, Ferrous & Ferric (Cont'd)	Matrix Spike	SM3500-Fe D	Frequency: 1 every 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag associated data outside of limit	—	N/A
	Matrix Spike Duplicate	SM3500-Fe D	Frequency: 1 every 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag associated data outside of limit	—	N/A
	Duplicate	SM3500-Fe D	N/A	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]	
Paint Filter	* Method Blank			9095A	N/A
	Laboratory Control Sample			9095A	N/A
	Matrix Spike			9095A	N/A
	Matrix Spike Duplicate			9095A	N/A
	Duplicate			9095A	Frequency: One per batch of 20 samples.
pH	* Method Blank	150.1 SM4500H-B	N/A	9040B 9040C904 5C 9041A	N/A
	Laboratory Control Sample	150.1 SM4500H- +B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9040B 9040C 9045C 9041	N/A
	Matrix Spike	150.1 SM4500H- +B	N/A	9040B 9040C 9045C 9041	N/A
	Matrix Spike Duplicate	150.1 SM4500H-B	N/A	9040B 9040C 9045C 9041	N/A
	Duplicate	150.1 SM4500H-B	Frequency: 1 with each batch of samples processed not to exceed 10 samples per matrix Criteria: ≤ 20 % RPD(3) limit Corrective Action: Flag data outside of limit.	9040B 9040C 9045C 9041	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are ≤ 20% RPD(3) Corrective Action Flag data associated with unacceptable Duplicate

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]	
Phenolics *	Method Blank	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Phenolics (Cont'd)	Laboratory Control Sample	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples
	Matrix Spike	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with unacceptable matrix spike	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike Duplicate	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]	
Phosphorus (Total and Ortho-phosphate)	* Method Blank	365.1 SM4500P-E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank		
	Laboratory Control Sample	365.1 SM4500P-E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples		
	Matrix Spike	365.1 SM4500P-E	Frequency: 1 per 20samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit		
	Matrix Spike Duplicate	365.1 SM4500P-E	Frequency: 1 per 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit		

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]	
Phosphorus (Total and Ortho-phosphate) (Cont'd)	Duplicate	365.1 SM4500P-E	N/A		
Solids	* Method Blank	160.1 160.2 160.3 160.4 160.5 SM2540C SM 2540D SM 2540F	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: If analyte level in method blank is ≥ RL for the analyte of interest in the sample, all associated samples with reportable levels of analyte are reprepared and reanalyzed.	—	N/A
	Laboratory Control Sample	160.1 160.2 160.3 160.4 160.5 SM2540C SM 2540D SM 2540F	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, reprepare and rerun all associated samples	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method RCRA (SW846) [2]
Solids (Cont'd)	Matrix Spike	160.1 160.2 160.3 160.4 160.5 SM2540C SM 2540D SM 2540F	N/A	—	N/A
	Matrix Spike Duplicate	160.1 160.2 160.3 160.4 160.5 SM2540C SM 2540D SM 2540F	N/A	—	N/A
	Duplicate	160.1 160.2 160.3 160.4 160.5 SM2540C SM 2540D SM 2540F	Frequency: 1 with each batch of samples processed not to exceed 10 samples Criteria: Sample results should agree within 20% if both the sample and sample duplicate results are > 5 X RL Corrective Action: Flag data outside of limit	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Sulfate	* Method Blank	375.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	9038	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank
	Laboratory Control Sample	375.4	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9038	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within ± 15 % Corrective Action: Rerun all samples associated with unacceptable LCS

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Sulfate (cont'd)	Matrix Spike	375.4	Frequency: 1 per 10 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9038	Frequency: 1 with each batch of samples processed not to exceed 10 samples Criteria: Limits are 75% - 125% recovery Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	375.4	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9038	N/A
	Duplicate	375.4	N/A	9038	N/A
Sulfide	* Method Blank	376.1 SM4500 S2 E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	9030B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Sulfide (Cont'd)	Laboratory Control Sample	376.1 SM4500 S2 E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9030B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike	376.1 SM4500 S2 E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9030B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data
	Matrix Spike Duplicate	376.1 SM4500 S2 E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9030B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag associated data Method 9034: Not Applicable
	Duplicate	376.1 SM4500 S2 E	N/A	9030B	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Total Organic Carbon (TOC)	* Method Blank	415.1 SM5310C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples. Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank
	Laboratory Control Sample	415.1 SM5310C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Method 9060 requires and LCS every 15 samples. Criteria: percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	415.1 SM5310C	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples. Method 9060 requires a matrix spike every 10 samples. Criteria: Percent recovery must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable Matrix Spike

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Total Organic Carbon (TOC) (cont'd)	Matrix Spike Duplicate	415.1 SM5310C	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	9060 Walkley-Black	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: Reanalyze if sample remaining. If not, flag data associated with unacceptable Matrix Spike Duplicate
	Duplicate	415.1 SM5310C	N/A	9060 Walkley-Black	Not Applicable

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Turbidity	* Method Blank	180.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	—	N/A

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Turbidity (cont'd)	Laboratory Control Sample	180.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	180.1	N/A	—	N/A
	Matrix Spike Duplicate	180.1	N/A	—	N/A
	Duplicate	180.1	Frequency: 1 per 10 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag data outside of limit Not Applicable.	—	N/A
Specific Gravity	* Method Blank	SM2710F	N/A		

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
	Laboratory Control Sample	SM2710F	N/A		
	Matrix Spike	SM2710F	N/A		
	Matrix Spike Duplicate	SM2710F	N/A		

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
	Duplicate	SM2710F	Frequency: One per batch of 20 samples.		
Mercury by CVAA & CVAFS	* Method Blank	245.1 1631E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank, unless the method blank is above RL, and samples are ND.	7470A 7471A 7471B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank, unless the method blank is above RL and samples are ND.

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Mercury by CVAA & CVAFS (Cont'd)	Laboratory Control Sample	245.1 1631E	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria:For 245.1 percent recovery of analyte must be within ± 20 %. For 1631E the percent recovery is +/- 23% Corrective Action: Rerun all samples associated with unacceptable LCS, unless samples are ND, results are reported.	7470A 7471A 7471B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria:percent recovery of analyte must be within ± 20 % Corrective Action: Rerun all samples associated with unacceptable LCS samples are ND, results are reported. . Exception: If samples are ND, results are reported.
	Matrix Spike	245.1 1631E	Frequency: with each batch of samples processed not to exceed 20 samples. 1631E frequency is 1 in 10 samples, 71-125% Criteria: For Method 245.1 recovery should be within 70-130 % Corrective Action: Flag data associated with unacceptable MS.	7470A 7471A 7471B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: For Method 7470A, recovery should be within 75-125 % . For Methods 7471A and 7471B, criteria is 70-130%. Corrective Action: Flag data associated with unacceptable MS.
	Matrix Spike Duplicate	245.1 1631E	Frequency: 1 with each batch of samples processed not to exceed 20 samples. 1631E frequency is 1 in 10 samples, 71-125% RPD 24% Criteria: For Method 245.1 recovery should be within 70-130 % Corrective Action: Flag data associated with unacceptable MSD	7470A 7471A 7471B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: For Method 7470A, recovery should be within 75-125 % , RPD[(3)] should be within 20%. . For Methods 7471A and 7471B, criteria is 70-130%. Corrective Action: Flag data associated with unacceptable MSD

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Mercury by CVAA & CVAFS (Cont'd)	Duplicate	245.1 1631E	N/A	7470A 7471A 7471B	N/A
ICP Metals	* Method Blank	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit. Concentration less than reporting with the exception of lab common contaminants. Sample results <RL are also valid. Corrective Action: Rerun all samples associated with unacceptable method blank unless the method blank is above RL, and samples are ND.	6010B 6010C 6020 6020A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit. Concentration less than reporting with the exception of lab common contaminants. Sample results <RL are also valid. Corrective Action: Rerun all samples associated with unacceptable method blank unless the method blank is above RL, and samples are ND.
	Laboratory Control Sample	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be ± 85-115%. If LCS is biased high and samples are <RL, the results are valid. Corrective Action: Rerun all samples associated with unacceptable LCS	6010B 6010C 6020 6020A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery of analyte must be ± 20 %. If LCS is biased high and samples are <RL, the results are valid. Corrective Action: Rerun all samples associated with unacceptable LCS If samples are ND, results are reported.

INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
ICP Metals (Cont'd)	Matrix Spike	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 70-130% Corrective Action: Flag data associated with unacceptable matrix spike	6010B 6010C 6020 6020A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery must be within laboratory limits. Corrective Action: Flag data associated with unacceptable matrix spike
	Matrix Spike Duplicate	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are 70-130%, RPD(3) must be within 20% Corrective Action: Flag data associated with unacceptable matrix spike	6010B 6010C 6020 6020A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Limits for percent recovery are must be within laboratory limits.RPD(3) must be within 20% Corrective Action: Flag data associated with unacceptable matrix spike
	Duplicate	200.7 200.8	Not Applicable	6010B 6010C 6020 6020A	Not Applicable
	Serial Dilution	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: 10% difference. 10% difference only applied if sample results are >50 times MDL. Corrective Action: Flag data associated with unacceptable serial dilution	6010B 6010C 6020 6020A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: 10% difference. 10% difference only applied if sample results are >50 times MDL. Corrective Action: Flag data associated with unacceptable serial dilution

Footnotes

[1] National Pollutant Discharge Elimination System

[2] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996), Update IV (2007).

[3] RPD-Relative Percent Difference

[4] Method not listed in 40 CFR Part 136. Method 300.0 is a proposed 40CFR method. Specific state and/or region approval is required for NPDES.

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis QC Sample Method		NPDES [1]	Method	RCRA (SW846) [2]
Herbicides Laboratory Control Sample			8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte must be within laboratory control limits Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS
Matrix Spike			8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte should be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike
Matrix Spike Duplicate			8151A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory control limits Corrective Action: Flag data associated with unacceptable matrix spike sample

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis QC	Sample Method		NPDES [1]	Method	RCRA (SW846) [2]
Herbicides (cont'd)	Duplicate			8151A	N/A
	Surrogates			8151A	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must fall within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards			8151A Optional	
Pesticides/ PCBs	* Method Blank	608	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	8081A 8081B 8082 8082A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Reprepare and reanalyze all samples associated with unacceptable method blank

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis QC	Sample Method		NPDES [1]	Method	RCRA (SW846) [2]
Pesticides/ PCBs (Cont'd)	Laboratory Control Sample	608	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery must be within control limits given in method for each analyte Corrective Action: Rerun all samples associated with unacceptable LCS	8081A 8081B 8082 8082A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	608	Frequency: 1 per 10 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8081A 8081B 8082 8082A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Pesticides/ PCBs (cont'd)	Matrix Spike Duplicate	608	N/A	8081A 8081B 8082 8082A	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	608	N/A	8081A 8081B 8082 8082A	N/A
	Surrogates	608	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: Results must fall within laboratory established control limits Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria	8081A 8081B 8082 8082A	Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: Results must fall within laboratory established control limits Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
Petroleum Hydro-carbons	* Method Blank	1664A	Frequency: 1 with each preparation batch Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	9071B	Frequency: 1 with each preparation batch Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis QC	Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Petroleum Hydro-carbons (Cont'd)	Laboratory Control Sample	1664A	Frequency: 1 with each analytical batch Criteria: Percent recovery is specified by the method, 78-114%, 11% RPD for HEM and 64-132%, 28% RPD for SGT HEM. Soils - Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS	9071B	Frequency: 1 with each analytical batch Criteria: Soils - Percent recovery must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS
	Matrix Spike	1664A	Frequency: 1 with every 10 samples per site Criteria: Percent recovery is specified by the method, 78-114%, 11% RPD for HEM and 64-132%, 28% RPD for SGT HEM Corrective Action: Corrective Action: Flag data associated with unacceptable Matrix Spike	9071B	Frequency: 1 with every 10 samples per site Criteria: Percent recovery must be within laboratory control limits Corrective Action:Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	1664A	NA	9071B	Frequency: 1 with every 10 samples per site Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike

Analysis QC	Sample Method		NPDES [1]	Method	RCRA (SW846) [2]
	Duplicate	1664A	N/A	9071B	N/A

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Semivolatiles	Matrix Spike	625	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8270C 8270D	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	625	N/A	8270C 8270D	Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Semivolatiles (Cont'd)	Duplicate	625	N/A	8270C 8270D	N/A
	Surrogates	625	Surrogates spiked into method blank and all samples (QC included) Method Blank and LCS Criteria: All surrogates must be in control before sample analysis may proceed. One surrogate per fraction may exceed control limits if greater than 10% recovery. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8270C 8270D	Surrogates spiked into method blank and all samples (QC included) Method Blank and LCS Criteria: All surrogates must be in control before sample analysis may proceed. One surrogate per fraction may exceed control limits if greater than 10% recovery. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	625	Frequency: ____ Internal standards spiked into method blank and all samples (QC included) Criteria: All internal standard recoveries must be within laboratory control limits Corrective Action: Flag sample data not meeting internal standard recovery requirements	8270C 8270D	Internal Standards are added to all samples (QC samples included). Internal standard area of daily standard must be within 50% to 200% of the response in the mid level of the initial calibration standard. The retention time (RT) for any internal standard (IS) in the continuing calibration must not exceed ± 0.5 minutes from mid level initial calibration standard IS RT.

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Volatiles by GC/MS	* Method Blank	624	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank	8260B 8260C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank
	Laboratory Control Sample	624	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8260B 8260C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte must be within laboratory control limits Corrective Action: Rerun all samples associated with unacceptable LCS

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis QC	Sample Method		NPDES [1]	Method	RCRA (SW846) [2]
Volatiles by GC/MS (Cont'd)	Matrix Spike	624	Frequency: 1 per ≤ 20 samples from each site or 1 per month, whichever is more frequent Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike	8260B 8260C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	624	N/A	8260B 8260C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory control limits Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	624	N/A	8260B 8260C	N/A

ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

Analysis QC	Sample Method	NPDES [1]	Method	RCRA (SW846) [2]
Volatiles by GC/MS (cont'd)	Surrogates	624 Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria	8260B 8260C	Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	624 Frequency: Internal standards spiked into method blank and all samples (QC included) Criteria: All internal standard recoveries must be within laboratory control limits Corrective Action: Flag sample data not meeting internal standard recovery requirements	8260B 8260C	Internal Standards are added to all samples (QC samples included). Internal standard area of daily standard must be within 50% to 200% of the response in the mid level of the initial calibration standard. The retention time (RT) for any internal standard (IS) in the continuing calibration must not exceed ± 0.5 minutes from mid level initial calibration standard IS RT.

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Methyl Mercury	* Method Blank	EPA 1630	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank		
	Laboratory Control Sample	EPA 1630	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike		
	Matrix Spike	EPA 1630	Frequency: 1 per ≤ 10 samples. Criteria: percent recovery for each analyte should be within laboratory limits. Corrective Action: Flag data associated with unacceptable Matrix Spike		
	Matrix Spike Duplicate	EPA 1630	Frequency: 1 per ≤ 10 samples. Criteria: percent recovery for each analyte should be within laboratory limits. Corrective Action: Flag data associated with unacceptable Matrix Spike		

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Formaldehyde	Duplicate	EPA 1630	N/A		
	Surrogates	EPA 1630	Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria		
	Method Blank			8315A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank
	Laboratory Control Sample			8315A	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Diesel Range Organics	Matrix Spike			8315A	Frequency: 1 per ≤ 20 samples. Criteria: percent recovery for each analyte should be within laboratory limits. Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate			8315A	Frequency: 1 per ≤ 10 samples. Criteria: percent recovery for each analyte should be within laboratory limits. Corrective Action: Flag data associated with unacceptable Matrix Spike
	Method Blank			8015B 8015C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank

Analysis QC	Sample Method		NPDES [1]	Method	RCRA (SW846) [2]
	Laboratory Control Sample			8015B 8015C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike			8015B 8015C	Frequency: 1 per ≤ 20 samples. Criteria: percent recovery for each analyte should be within laboratory limits. Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate			8015B 8015C	Frequency: 1 per ≤ 10 samples. Criteria: percent recovery for each analyte should be within laboratory limits. Corrective Action: Flag data associated with unacceptable Matrix Spike
	Surrogates			8015B 8015C	Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Gasoline Range Organics	Method Blank			8015B 8015C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank
	Laboratory Control Sample			8015B 8015C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike			8015B 8015C	Frequency: 1 per ≤ 20 samples. Criteria: percent recovery for each analyte should be within laboratory limits. Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate			8015B 8015C	Frequency: 1 per ≤ 10 samples. Criteria: percent recovery for each analyte should be within laboratory limits. Corrective Action: Flag data associated with unacceptable Matrix Spike

Analysis	QC Sample	Method	NPDES [1]	Method	RCRA (SW846) [2]
Aromatic Acids	Surrogates			8015B 8015C	Surrogates spiked into Method Blank and all samples (QC included) Method Blank Criteria: All surrogates must be in control before sample analysis may proceed. Sample Criteria: Re-extract samples or flag sample data not meeting surrogate criteria
	Method Blank			SOP Frequency: 1 with each batch of samples processed not to exceed 20 samples	Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank
	Laboratory Control Sample			SOP Frequency: 1 with each batch of samples processed not to exceed 20 samples	Criteria: percent recovery for each analyte should be within advisory limits given in method Corrective Action: Flag data associated with unacceptable Matrix Spike
	Matrix Spike			SOP	Frequency: 1 per ≤ 20 samples. Criteria: percent recovery for each analyte should be within laboratory limits. Corrective Action: Flag data associated with unacceptable Matrix Spike

Analysis QC	Sample Method		NPDES [1]	Method	RCRA (SW846) [2]
	Matrix Spike Duplicate			SOP	Frequency: 1 per ≤ 10 samples. Criteria: percent recovery for each analyte should be within laboratory limits. Corrective Action: Flag data associated with unacceptable Matrix Spike

Footnotes

[1] National Pollutant Discharge Elimination System

[2] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IV (2007)

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SECTION 13

PREVENTIVE ACTION / IMPROVEMENT

13.1 OVERVIEW

The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and client satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the monthly QA Metrics Report, evaluation of internal or external audits, results and evaluation of proficiency testing (PT) performance, data analysis and review processing operations, client complaints, staff observation, etc.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, Ethics training, etc. These metrics are used in evaluating themanagement and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process (Section 13) is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process, and management review.

13.1.2 Any Preventive Actions undertaken or attempted must be taken into account during the Annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of success and failure within the preventive action program is sufficient to provide management with a measurement for evaluation.

13.2 MANAGEMENT OF CHANGE

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The laboratory has a graded approach for managing change based based on the Management Systems Review.

SECTION 14

CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

14.1 OVERVIEW

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the QA Department which is backed up as part of the regular network backup. Records are of two types--either electronic or hard-copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by the Records Manager.

Table 14-1. Records Index [1]

	Record Types [1]:	Retention Time:
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals	5 Years from document retirement date*
QA Records	- Internal & External Audits/Responses - Certifications - Corrective/Preventive Actions - Management Reviews - Method & Software Validation / Verification Data - Data Investigation	5 Years from archival* Data Investigation: 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
Project Records	- Sample Receipt & COC Documentation - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*

	Record Types [1]:	Retention Time:
Administrative Records	Finance and Accounting	10 years
	EH&S Manual, Permits	7 years
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	7 Years (HR Personnel Files must be maintained indefinitely)
	Administrative Policies Technical Training Records	7 years

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 14-2.

All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility that provides a suitable environment to prevent damage or deterioration and to prevent loss . All records must be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records and electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees, and shall be documented with an access log. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention must be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

14.1.1 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Note: For the Ohio VAP program the laboratory is required to notify Ohio EPA of its intent to dispose of any records.

Table 14-2. Special Record Retention Requirements

Program	Retention Requirement
Ohio – Drinking Water	5 years (project records) 10 years – radio chemistry (project records)
Michigan Department of Environmental Quality – all environmental data	10 years
OSHA - 40 CFR Part 1910	30 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement and others as negotiated.
Ohio Voluntary Action Program	10 years

Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

14.1.2 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hardcopy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.14.1 for more information.

14.1.3 The record-keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. (Records stored off site should be accessible within two days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This must include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory copy of the Chain-of-Custody is stored with the invoice and the Work Order sheet generated by LIMS. The Chain-of-Custody would indicate the name of the sampler. If any sampling notes are provided with a Work Order, they are kept with this package.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The record-keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes, e.g., set format for naming electronic files, set format for what is included with a given analytical data set. SOP NC-QA-019, Records Information Management, outlines this procedure. Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; each day's run long or instrument sequence

is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is entered into LIMS for each method as required.

- Changes to hardcopy records must follow the procedures outlined in Sections 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "Sampled by," "Prepared by," "Reviewed by", or "Analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard-copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure no data is lost, and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard-copy which was scanned.
- Also refer to Section 19.14.1, "Computer and Electronic Data Related Requirements".

14.2 TECHNICAL AND ANALYTICAL RECORDS

14.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement (refer to Section 15.1). The records for each analysis must contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records must include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing results.

14.2.2 Observations, data, and calculations are recorded in real-time at the time they are made and are identifiable to the specific task.

14.2.3 Changes to hardcopy records must follow the procedures outlined in Sections 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails. The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- Laboratory sample ID code
- Date of analysis. Time of analysis is also required if the holding time is 72 hours or less, or when time-critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available. Instrument logs may be in electronic format.

- Analysis type
- All manual calculations and manual integrations
- Analyst or operator initials/signature
- Sample preparation, including cleanup, separation protocols, incubation periods ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents
- Test results
- Standard and reagent origin, receipt, preparation, and use
- Calibration criteria, frequency, and acceptance criteria
- Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions
- Quality control protocols and assessment
- Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.

14.3 LABORATORY SUPPORT ACTIVITIES

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- All original raw data, whether hard-copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records)
- A written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value
- Copies of final reports
- Archived SOPs
- Correspondence relating to laboratory activities for a specific project
- All Corrective Action reports, audits and audit responses
- Proficiency test results and raw data
- Results of data review, verification, and cross-checking procedures

14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include, but are not limited to, records pertaining to:

- Sample preservation including appropriateness of sample container and compliance with

holding time requirement

- Sample identification, receipt, acceptance or rejection and login
- Sample storage and tracking including shipping receipts, sample transmittal / COC forms
- Procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

14.4 ADMINISTRATIVE RECORDS

The laboratory also maintains the administrative records in either electronic or hard-copy form Refer to Table 14-1.

14.5 RECORDS MANAGEMENT, STORAGE, AND DISPOSAL

All records (including those pertaining to test equipment), certificates, and reports are safely stored, held secure, and in confidence to the client. Certification-related records are available to the accrediting body upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hardcopy, write-protected backup copies, or an electronic audit trail controlling access.

The laboratory has a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage, and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially.

14.6 TRANSFER OF OWNERSHIP

In the event the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous five years of such action.

14.7 RECORDS DISPOSAL

Records are removed from the archive and destroyed after five years, unless otherwise specified by a client or regulatory requirement. On a project-specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that

ensures their confidentiality such as shredding, mut ilation or incineration (refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15

AUDITS

15.1 INTERNAL AUDITS

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Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and when requested to corporate management.

Audits are conducted and documented as described in TestAmerica Corporate SOP CA-Q-S-004 on performing Internal Auditing. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or Corporate QA	All areas of the laboratory annually
Method Audits	Joint responsibility: a) QA Manager or designee with assistance by the Technical Director or designee (refer to CA-Q-S-004)	Method audits frequency: 50% of methods annually 100% of methods annually (DoD Labs)
-		
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI field of testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, TNI quality systems , client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to, data review, quality controls, preventive action, and corrective action. The completeness of earlier corrective action is assessed for effectiveness and sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

Note: Part of the quality systems audit relates to regulatory compliance. An assessment of the laboratory's compliance to regulatory requirements is performed by Corporate QA through monthly management reports, review of client and regulatory concerns and also through periodic on-site evaluations.

15.1.2 QA Technical Audits

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit Miner programs (e.g., Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits must include all methods within a two-year period.

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs must be assessed by the Technical Director and the QA department at least every two years. The work of each newly hired analyst is assessed within three months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products must be performed within three months of completing the documented training.

15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

15.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the followi ng types of PT studies--nonpotable water and soil

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.2 EXTERNAL AUDITS

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory group leaders are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the laboratory's Corrective Action plan must be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 TNI standards.

15.3 AUDIT FINDINGS

Audit findings are documented using the Corrective Action process and spreadsheet. The laboratory's Corrective Action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by Operations management and the QA Manager.

Developing and implementing Corrective Action to fin dings is the responsibility of the Department Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the laboratory's Corrective Action plan must be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory must take timely corrective action, and must notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24 hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

SECTION 16

MANAGEMENT REVIEWS

16.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report must be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director and their Corporate Quality Director as well as the General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager, or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

16.2 ANNUAL MANAGEMENT REVIEW

The Senior Lab Management Team (Laboratory Director, Technical Director, Operations Manager, QA Manager, HR Supervisor, PM Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives, and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory must summarize any critical findings that cannot be solved by the lab, and report them to Corporate IT.

The Management Systems Review (Corporate SOP CA-Q-S-008 and Work Instruction CA-Q-WI-020) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective; therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review
- Prior Monthly QA Reports issues
- Laboratory QA Metrics
- Review of report reissue requests
- Review of client feedback and complaints
- Issues arising from any prior management or staff meetings

- Minutes from prior Senior Lab Management Team meetings. Issues that may be raised from these meetings include:
  - Adequacy of staff, equipment and facility resources
  - Adequacy of policies and procedures
  - Future plans for resources and testing capability and capacity
- The annual internal double blind PT program sample performance (if performed)
- Compliance to the Ethics Policy and Data Integrity Plan, including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and Corporate Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants
- A reference to the existing data quality related documents and topics that were reviewed
- Quality system or operational changes or improvements that will be made as a result of the review, e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)

Changes to the quality systems requiring update to the laboratory QA Manual must be included in the next revision of the QA Manual.

16.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/ Recall SOP CW-L-S-002must be followed. All investigations that result in finding inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's COO, VP of Client & Technical Services, General Managers and Corporate Quality Directors receive a monthly report from the Director of Quality & Client Advocacy summarizing any current data integrity or data recall investigations. The General Managers are also made aware of progress on these issues for their specific labs.

SECTION 17

PERSONNEL

17.1 OVERVIEW

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training must have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff must be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training must be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL PERSONNEL

The laboratory makes every effort to hire analytical staff that posses a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. There are competent analysts and technicians in the industry who have not earned a college degree. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all pe rsonnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet "Human Resources" web-page (also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance or quantitation techniques, etc. are also considered

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least one year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	Or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience. Or 5 years of prior analytical experience
Group Leaders – General	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Group Leader – Wet Chem only (no advanced instrumentation)	Associate degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Department Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.3 TRAINING

The laboratory is committed to furthering the profes sional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame*	Employee Type
New Hire Orientation	Immediately	All
Environmental Health & Safety Orientation	Prior to lab work	All
Environmental Health & Safety Orientation Follow-up Test	30-60 days after hire All	
Environmental Health & Safety Training Refer to EH&S Manual All		
Ethics – New Hires	1 week of hire	All
Ethics - Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC) Prior to unsupervised method performance		Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in the employee's training file.
- Documentation of proficiency (refer to Section 19)
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training
- A Confidentiality Agreement signed by each staff member signed at the time of employment
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct, e.g., ethics. This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts' knowledge to refer to QA Manual for quality issues
- Analysts following SOPs, i.e., practice matches SOPs
- Analysts regularly communicate to group leaders and QA if SOPs need revision rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the Laboratory Training SOP NC-QA-028, Employee Orientation and Training.

17.4 DATA INTEGRITY AND ETHICS TRAINING PROGRAM

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within one week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and annual refresher for all employees. Senior management at each facility performs the Ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times, TestAmerica has established a Corporate Ethics Policy (CW-L-P-004) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement/Agreement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts; and for that reason, TestAmerica has a zero tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping
- Discussion regarding data integrity procedures

- Specific examples of breaches of ethical behavior--peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion
- Internal monitoring. Investigations and data recalls
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient

Additionally, a Data Integrity Hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 18

ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

18.1 OVERVIEW

The laboratory is a 54,440 sq. ft. secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity-controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

18.2 ENVIRONMENT

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. A 225KVA UPS is installed in the main electrical bus to provide at least 15 minutes of backup power in the event of a power failure. This unit also provides voltage and frequency control of lab and office power. A spike/surge arrestor is installed to protect against power surge/sag and lightning strikes. A 30 KW natural gas-fueled backup generator is installed to provide power to the I.T. area in the event of a power failure. Additionally, this generator provides power to two walk-in sample

storage coolers and several other smaller sample sto rage coolers. Smaller portable generators are available to provide "spot power" where needed in the event of a power failure.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing must be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

18.3 WORK AREAS

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to, and use of, all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory
- Sample receipt areas
- Sample storage areas
- Chemical and waste storage areas
- Data handling and storage areas
- Sample processing areas
- Sample analysis areas

18.4 FLOOR PLAN

A floor plan can be found in Appendix 1.

18.5 BUILDING SECURITY

Building keys are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into

the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

Signs are posted in the laboratory designating employee only areas - "Authorized employees beyond this point".

SECTION 19

TEST METHODS AND METHOD VALIDATION

19.1 OVERVIEW

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples; and where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 STANDARD OPERATING PROCEDURES (SOPs)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP CW-Q-S-002 entitled Writing a Standard Operating Procedure, or the laboratory's SOP NC-QA-027, Preparation and Management of Standard Operating Procedures.
- SOPs are reviewed at a minimum of every two years (annually for DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 LABORATORY METHODS MANUAL

For each test method, the laboratory must have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory must demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both techn ical and non-technical SOPs . Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 SELECTION OF METHODS

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services, e.g., special matrices, non-routine compound lists, etc., the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods must be used.

When clients do not specify the method to be used or methods are not required, the methods used must be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, February 1999*
- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th edition/ on-line edition Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*

- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996, Final Update IV, January 2008.*

- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*

- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory must inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it must be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory must confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability is performed (SOP NC-QA-028, Employee Orientation and Training) whenever there is a change in instrument type (e.g., new instrumentation), method, or personnel (e.g., analyst has not performed the test within the last 12 months).

The initial demonstration of capability must be thoroughly documented and approved by the Technical Director and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve (low standard at or below the QL)and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).

  Note: For Ohio VAP work, the term Reporting Limit will be used.

- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted as " *Reporting Limit based on the low standard of the calibration curve".*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

At least four aliquots must be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest. Refer to SOP NC-QA-028, Employee Orientation and Training, for details on this procedure.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (see Figure 19-1 as an example) must be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

19.5 LABORATORY-DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 VALIDATION OF METHODS

Validation is the confirmation by examination and th e provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

19.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch-specific QC samples such as LCS, method blanks, or PT samples.

19.7 METHOD DETECTION LIMITS (MDL)/ LIMITS OF DETECTION (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B, or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to Section 19.7.10). Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. To allow for some flexibility, this low level standard may be analyzed every batch or every week or some other frequency rather than doing the study all at once. In addition, a larger number of data points may be used if the appropriate t-value multiplier is used.

Refer to the Corporate SOP CA-Q-S-006 or the laboratory's SOP NC-QA-021 for details on the laboratory MDL process.

Note: For Ohio VAP projects, the MDL procedure must also comply with OAC Rule 3745-300-01(A)(78).

19.8 INSTRUMENT DETECTION LIMITS (IDL)

The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either by using seven replicate spike analyses, like MDL but without sample preparation, or by the analysis of ten instrument blanks and calculating three times the absolute value of the standard deviation.

If IDL is > than the MDL, it may be used as the reported MDL.

19.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDLV standard is not successful, then the laboratory will redevelop their MDL or perform and pass two consecutive MDLVs at a higher concentration and set the LOD at the higher concentration. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation. Refer to the laboratory SOP NC-QA-021 or Corporate CA-Q-S-006 for further details.

The laboratory quantitation limit is equivalent to the DoD Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects. For DoD projects, TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ. The RL is a

level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

19.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept in each department. Complete details are available in the laboratory SOPs.

19.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, atomic absorption, or fluorescence profiles.

19.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

19.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.12.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The

percent recovery of the LCS is compared either to th e method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 $\pm$ 0.5 mg/l.

19.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement, e.g., 524.2, 525, etc., and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.13 SAMPLE REANALYSIS GUIDELINES

19.13.1 Because there is a certain level of uncertainty with any analytical measurement, a sample repreparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. **Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.**

- Homogenous samples: If a re-analysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within + _ 1 reporting limit for samples < _ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the re-analysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.

- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.

- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Area Supervisor or Laboratory Director if unsure.

19.14 CONTROL OF DATA

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.14.1 Computer and Electronic Data Related Require ments

The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently running the TALS LIMS which is a custom in-house developed LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes **Microsoft SQL, which is a relational database platform.** It is referred to as Database for the remainder of this section.

19.14.1.1 Maintain the Database Integrity

Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails, and contorlled access.

19.14.1.2 Ensure Information Availability

Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.14.1.3 Maintain Confidentiality

Ensure data confidentiality through physical access controls, such as password protection or website access approval, when electronically transmitting data.

19.14.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved, e.g., extractions, dilutions, instrument readings and concentrations. The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by peer review once updated in LIMS. The review checklists are signed by both the analyst and reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP CA-Q-S-002, Acceptable Manual Integration Practices.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it must not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.14.2.1 All raw data must be retained. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.14.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. The units "mg/l" and "mg/kg" are the same as "parts per million (ppm)". The units " µg/l" and " µg/kg" are the same as "parts per billion (ppb)." For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1%. Units are defined in each lab SOP.

19.14.2.3 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

19.14.2.4 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst reviews what has been entered to check for errors.

19.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out in 'real time' and have enough information on them to trace the events of the applicable analysis/task (e.g., calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.).

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA Department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

19.14.4 Review / Verification Procedures

19.14.4.1 Data Recording Procedures

To ensure data integrity, all documentation of data and records generated or used during the process of data generation must be performed in compliance with Section 3 of this document and Policy CA-Q-T-005, Laboratory Documentation.

19.14.4.2 Data Reduction and Verification Procedures

Data review procedures comprise a set of computerized and manual checks applied at appropriate levels of the measurement process. Data review begins with the reduction or processing of data and continues through verification of the data and the reporting of analytical results. Calculations are checked from the raw data to the final value prior to reporting results for each group of samples. Data reduction can be performed by the analyst who obtained the data or by another analyst. Data verification starts with the analyst who performs a 100% review of the data to ensure the work was done correctly the first time. Data verification continues with review by a second reviewer who verifies that data reduction has been correctly performed and that the analytical results correspond to the data acquired and processed.

19.14.4.2.1 Data Reduction and Initial Verification

Data reduction and initial verification may be performed by more than one analyst depending upon the analytical method employed. The preparation and analytical data may be reviewed independently by different analysts. In these instances, each item may not be applicable to the subset of the data verified or an item may be applicable in both instances. It is the responsibility of the analyst to ensure that the verification of data in his or her area is complete. The data reduction and initial verification process must ensure that:

- Sample preparation information is correct and complete including documentation of standard identification, solvent lot numbers, sample amounts, etc.
- Analysis information is correct and complete including proper identification of analysis output (charts, chromatograms, mass spectra, etc.)
- Analytical results are correct and complete including calculation or verification of instrument calibration, QC results, and qualitative and quantitative sample results with appropriate qualifiers
- The appropriate SOPs have been followed and are identified in the project and/or laboratory records
- Proper documentation procedures have been followed
- All non-conformances have been documented
- Special sample preparation and analytical requirements have been met.
- The data generated have been reported with the appropriate number of significant figures as defined by the analytical method in the LIMS or otherwise specified by the client.

In general, data will be processed by an analyst in one of the following ways:

- Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets
- Input of raw data for computer processing
- Direct acquisition and processing of raw data by a computer.

If data are manually processed by an analyst, all steps in the computation must be provided including equations used and the source of input parameters such as response factors (RFs), dilution factors, and calibration constants. If calculations are not performed directly on the data sheet, they may be attached to the data sheets.

Manual integrations are sometimes necessary to correct misintegrations by an automatic data system software program, but must only be performed when necessary. Further discussion of manual integrations and the required documentation is given in Policy CA-Q-S-002, Acceptable Manual Integration Practices.

For data that are input by an analyst and processed using a computer, a copy of the input must be kept and uniquely identified with the project number and other information as needed. The samples analyzed must be clearly identified.

If data are directly acquired from instrumentation or a test procedure and processed, or immediately entered into LIMS, the analyst must verify that the following are correct:

- Project and sample numbers
- Calibration constants and RFs
- Units
- Numerical values used for reporting limits.

Analysis-specific calculations for methods are provided in SOPs. In cases where computers perform the calculations, software must be validated or verified, as described in Section 6.0 of this document, before it is used to process data.

The data reduction is documented, signed and dated by the analyst completing the process. Initial verification of the data reduction by the same analyst is documented on a data review checklist, signed and dated by the analyst.

19.14.4.2.2 Data Verification

Following the completion of the initial verification by the analyst performing the data reduction, a systematic check of the data that has been fully reduced and checked through Level 1 review is performed by an experienced peer, group leader, or designee. This Level 2 check is performed to ensure that Level 1 review has been completed correctly and thoroughly. The second level reviewer examines the data signed by the analyst. Any exceptions noted by the analyst must be reviewed. Included in this review is an assessment of the acceptability of the data with respect to:

- Adherence of the procedure used to the requested analytical method SOP
- Correct interpretation of chromatograms, mass spectra, etc.
- Correctness of numerical input when computer programs are used (checked randomly)
- Correct identification and quantitation of constituents with appropriate qualifiers
- Numerical correctness of calculations and formulas (checked randomly)

- Acceptability of QC data (100% review)
- Documentation that instruments were operating according to method specifications (calibrations, performance checks, etc.)
- Documentation of dilution factors, standard concentrations, etc.
- Sample holding time assessment.

This review also serves as verification that the process the analyst has followed is correct in regard to the following:

- The analytical procedure follows the methods and client-specific instructions.
- Nonconforming events have been addressed by corrective action as defined on a nonconformance memo
- Valid interpretations have been made during the examination of the data and the review comments of the initial reviewer are correct
- The package contains all of the necessary documentation for data review and report production and results are reported in a manner consistent with the method used for preparation of data reports.

The specific items covered in the second stage of data verification may vary according to the analytical method, but this review of the data must be documented by signing the same checklist.

19.14.4.2.3 Completeness Verification

A third-level review is performed by the reporting and project management staff. This review is required before results are submitted to clients. This review serves to verify the completeness of the data report and to ensure that project requirements are met for the analyses performed. The items to be reviewed are:

- Analysis results are present for every sample in the analytical batch, reporting group, or sample delivery group (SDG)

- Every parameter or target compound requested is reported with either a value or reporting limit
- All nonconformances, including holding time violations, and data evaluation statements that impact the data quality are accompanied by clearly expressed comments from the laboratory

- The final report contains all the supporting documentation required by the project, and is in either the standard TestAmerica format or in the client-required format.

- Implement checks to monitor the quality of laboratory results using correlation of results for different parameters of a sample (for example, does the TOC results justify the concentration of organic compounds found by GC/MS.)

- A narrative to accompany the final report must be finalized by the PM. This narrative must include relevant comments collected during the earlier reviews.

The Quality Assurance Department performs data reviews per SOP CA-Q-S-004, Internal Auditing. For DoD work, 10% of all reports must undergo an internal data review.

19.14.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002).

19.14.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.14.5.2 Analysts must not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.

19.14.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.14.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate-approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1. Example - Demonstration of Capability Documentation

Analyst Demonstration of Capability
Certification Statement

Analyst Name
Date:

Test Method:
SOP:
Matrix:

TestAmerica North Canton laboratory
4101 Shuffel Drive NW
North Canton, OH 44720
(330) 497-9396

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the TestAmerica Quality Assurance Plan, has met the Initial or Ongoing Demonstration of Capability.

2. The test method was performed by the analyst identified on this certification following the TestAmerica SOP.

3. A copy of the laboratory-specific SOP is available for all personnel on-site.

4. The data associated with the initial/ongoing demonstration of capability are true, accurate, complete and self-explanatory (*). These data are attached to this certification statement.

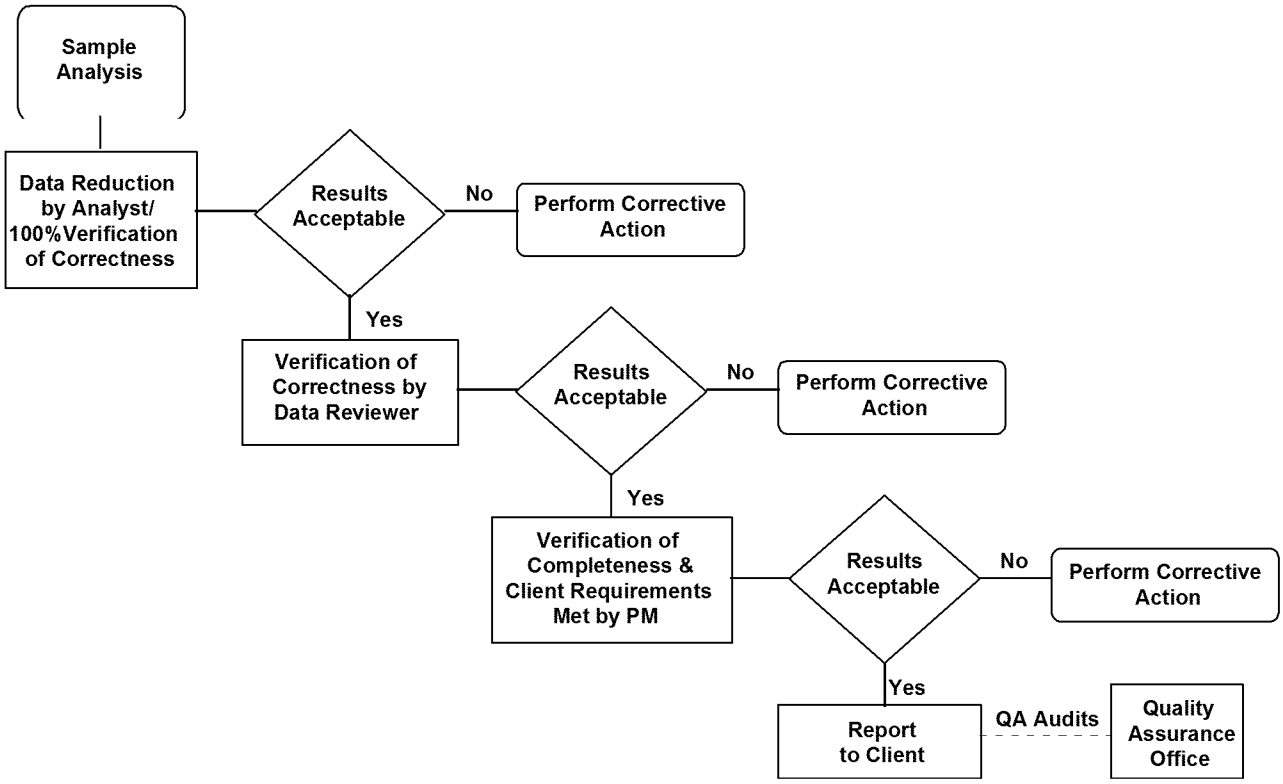
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

Comments/Observations:

Technical Director's Name	Signature	Date
QA Manager's Name	Signature	Date

* *True: Consistent with supporting data.*
Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.
Complete: Includes the results of all supporting performance testing.
Self-explanatory: Data properly labeled and stored so that the results are traceable and require no additional explanation.

Figure 19-2. Work Flow



SECTION 20

EQUIPMENT AND CALIBRATIONS

20.1 OVERVIEW

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory equipment and instrumentation is presented in Table 20-1

Equipment is only operated by authorized and trained personnel. Manufacturers instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 PREVENTIVE MAINTENANCE

20.2.1 The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

20.2.2 Routine preventive maintenance procedures and frequency, such as lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

20.2.3 Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Group Leader to ensure instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures are also be outlined in analytical SOPs or instrument manuals. (Note: For some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

20.2.4 Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs must be kept for all major pieces of equipment. Instrument Maintenance Logbooks may also be used to specify instrument parameters.

20.2.4.1 Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

20.2.4.2 Each entry in the instrument log includes the Analyst's initials, date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control, e.g., CCV run on 'date' was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

20.2.4.3 When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled-in page must be signed across the page entered and the logbook, so it is clear that a page is missing if only half a signature is found in the logbook.

20.2.5 Instrument Repair

If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it must be taken out of operation and tagged as out of service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory must examine the effect of this defect on previous analyses.

20.2.6 Equipment Malfunction

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Backup instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the backup is not available and the analysis cannot be carried out within the needed timeframe, the samples must be subcontracted.

20.2.7 Instrument Transfer or Send-Out

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

20.3 SUPPORT EQUIPMENT

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, dispensing devices, if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM Type 1 weights spanning its range of use (weights that have been calibrated to ASTM Type 1 weights may also be used for daily verification). ASTM Type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed. They are calibrated at least every five years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM Type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. Reference SOP NC-QA-015, Equipment Monitoring and Thermometer Calibration. A list of balances is in Table 21.2.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to + _ 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes, thermocouples, refrigerator thermometers (not NIST-Traceable), and freezer thermometers (not NIST –Traceable) are calibrated quarterly.

The NIST thermometer is recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logsheets. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logsheets. More information on this subject can be found in SOP NC-QA-015, Equipment Monitoring and Thermometer Calibration.

20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day (seven days a week for DOD labs).

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between or $4 \pm 2^{\circ}C$.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logsheets posted on each unit.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A glassware and glass microliter syringes) are given unique identification numbers and the deliveery volumes are verified gravimetrically, at a minimum, on a quarterly basis.
Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

The laboratory maintains a sufficient inventory of autopipettors, and dilutors of differing capacities that fulfill all method requirements.

These devices are given unique identification numbers, and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

Any device not regularly verified cannot be used for any quantitative measurements.

20.3.7 Field Sampling Devices (ISCO Autosamplers)

Each autosampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is recorded on the sampling documentation in a logbook.

The autosampler is calibrated semi-annually by setti ng the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The autosampler is programmed to run three cycles, and each of the three cycles is measured into a beaker to verify 100 ml are received.

If the RSD (Relative Standard Deviation) between the three cycles is greater than 20%, the procedure is repeated. If the result is still greater than 20%, the following options may be employed:

1) The unit is taken out of service.
2) The unit is used to pull composite samples only over a 24-hour period.

The results of this check are kept in a logbook/binder.

20.4 INSTRUMENT CALIBRATIONS

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, action is performed and any affected samples are re-analyzed if possible. If the re-analysis is not possible, any data associated with an unacceptable initial calibration must be reported with appropriate data qualifiers (refer to Section 12). All sample analyses reported for Ohio VAP certified data must be associated with a valid calibration.

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.4.1 CALIBRATION STANDARDS

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of three calibration points (exception being ICP and ICP/MS methods) will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.2 Calibration Verification

The calibration relationship established during the initial calibration must be verified at initially (with an ICV) and at least daily (with a CCV) as specified in the laboratory method SOPs in accordance with the referenced analytical methods and and in the 2009 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

Note: The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 TNI Standard EL-V1M4 Section 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinataive methods or SOPs.

Note: If an internal standard calibration is being used (basically GCMS), then bracketing standards are not required. Only daily verifications are needed. The results from these

verification standards must meet the calibration veirfication criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after ever 10 samples or injections include matrix or batch QC samples.

Note: If an internal standard calibration is being used (basically GCMS), then bracketing standards are not required. Only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed and documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial isntrument calibration shall be performed.

Sample analyses and reporting of data my not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable under the following special conditions and reported based upon discussion and approval of the client.

a) When acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-dectects may be reported with a footnote or case narrative explaining the high bias. Otherwise, the samples affected by the unacceptable CCV shall be re-analyzed after a new calibraiton curve has been established, evaluated, and accepted; or

b) When the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated, and accepted.

Samples reported by the two conditions identified above will be appropriately flagged.

20.4.2.1 Verification of Linear Calibrations

Calibration verification for linear calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the

laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. For Ohio VAP samples, results may not be reported when calibration verifications are exceeded low.

20.5 TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. TICs cannot be reported as "VAP certified" data for Ohio VAP projects.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

20.6 GC/MS TUNING

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any

adjustment but it may be required based on the curre nt instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Laboratory Equipment and Instrumentation

Instrument Type	Manufacturer	Model/Serial No.	Year into Service
GC/MS Volatiles	Hewlett-Packard (UX2)	5971A-5890, S/N 3140A38490 (screening)	1992
	Hewlett-Packard (HP6)	5973-6890, S/N US00005571 (screening)	1998
	Hewlett-Packard (UX7)	5973-6890, S/N US00010937 (screening)	1998
	Hewlett-Packard (UX8)	5973-6890, S/N US00027773	1999
	Hewlett-Packard (UX9)	5973-6890, S/N US00028329	2000
	Hewlett-Packard (UX10)	5973-6890, S/N US00032072	2000
	Agilent (UX11)	5973-6890, S/N US00038093	2000
	Agilent (UX12)	5973-6890, S/N US10202133	2002
	Agilent (UX14)	5973-6890, S/N CN10340027	2003
	Agilent (UX15)	5973-6890, S/N CN10515062	2005
	Agilent (UX16)	5975-6890, S/N CN10539065	2005
GC/MS Volatiles Autosampler	OI Analytical (UX2)	4552, S/N 12596 (screening)	1999
	OI Analytical (HP6)	4552, S/N 12258 (screening)	1998
	OI Analytical (UX7)	4552, S/N 13154 (screening)	1998
	OI Analytical (UX8)	4552, S/N 13089	1999
	OI Analytical (UX9)	4552, S/N 13233	2000
	OI Analytical (UX10)	4552, S/N 12058	2000
	OI Analytical (UX11)	4552, S/N 13408	2000
	OI Analytical (UX12)	4552, S/N 13667	2002
	OI Analytical (UX14)	4552, S/N 14092	2003
	OI Analytical (UX15)	4552, S/N 14368	2005
	OI Analytical (UX16)	4552, S/N 14519	2005
GC/MS Volatiles Purge and Trap	OI Analytical (UX2)	4560, S/N K822460889 (screening)	1999
	OI Analytical (HP6)	Encon (screening)	1998
	OI Analytical (UX7)	4660, S/N N251460461 (screening)	2004
	OI Analytical (UX8)	4560, S/N B444466152P	2004
	OI Analytical (UX9)	4560, S/N M946460832	2000
	OI Analytical (UX10)	4660, S/N BETA6	2003
	OI Analytical (UX11)	4560	2000
	OI Analytical (UX12)	4560, S/N NM041460393	2002
	OI Analytical (UX14)	4660	2008

Instrument Type	Manufacturer	Model/Serial No.	Year into Service
	OI Analytical (UX15)	4660, S/N C511466149P	2005
	OI Analytical (UX16)	4660, S/N D539446261P	2005
GC/MS Semivolatiles	Hewlett-Packard HP7	5973-6890, S/N US71190756-US00009247	1998
	Hewlett-Packard HP9	5973-6890, S/N US91422379-US72020889	2000
	Agilent HP10	5973-6890, S/N US33220074-CN10340002	2003
	Agilent A4AG2	5975C-7890, S/N US71235692-CN10721110	2007
GC Volatiles	Agilent (A)	6890 PID/FID, S/N US10402056	2004
	Hewlett-Packard (O)	6890 Dual PID/Hall, S/N US00007206	1997
	Hewlett-Packard (P)	6890 PID/HALL, S/N US00030616	1997
	Hewlett-Packard (Y)	6890N PID/FID, S/N US10337062	2003
GC Volatiles Auto Sampler	OI Analytical (O)	Archon, S/N 13196	2000
	OI Analytical (Y)	4552, S/N 14045	1998
	Varian (A)	Archon SN 12019	1998
	Varian (P)	4552	2000
GC Volatiles Purge & Trap	OI Analytical (O)	4560	2000
	Tekmar (P)	3000	1993
	Varian (A)	3000	1998
	Varian (Y)	3000	1993
GC Volatiles Purge & Trap			
GC Semivolatiles	Hewlett-Packard (P1)	6890 EPC & Dual ECD Y-Splitter S/N US00023208	1998
	Hewlett-Packard (P2)	6890 EPC & Dual ECD Y-Splitter S/N US00023512	1998

Instrument Type	Manufacturer	Model/Serial No.	Year into Service
	Hewlett-Packard (P3)	6890 EPC & Dual ECD Y-Splitter S/N US00023674	1998
	Hewlett-Packard (P4)	6890 EPC & Dual ECD Y-Splitter S/N US00029531	1999
	Hewlett-Packard (P5)	6890 EPC & Dual ECD Y-Splitter S/N US11911568	2010
	Hewlett-Packard (P6)	6890 EPC & Dual FID S/N US00032848	2000
	Agilent (P9)	6890N EPC & Dual ECD Y-Splitter S/N US10205045	2005
	Agilent (P10)	6890 EPC & Dual ECD Y-Splitter S/N US10151110	1999
	Agilent (P11)	6890N EPC & Dual ECD Y-Splitter S/N CN10517088	2004
	Agilent (P12)	6890N EPC & Dual ECD Y-Splitter S/N CN10512025	2005
	Agilent (P13)	6890N EPC & Dual ECD Y-Splitter S/N CN10435032	2004
	Agilent (P14)	7890 EPC & Dual FID S/N CN 10281044	2010
GC Semivolatiles HPLC	Hewlett-Packard (L2)	HPLC 1100, S/N US82404153	1998
Extractions Sonicators	Misonix	3000 (self-tuning), S/N R1044	2005
	Misonex	Ultrasonic Processor XL, S/N G4221	2005
	Heat Systems	XL2020, S/N G1026	2005
Extractions pH Meter	Thermo Orion	420, S/N 074028	1998
	Mettler Toledo	SevenEasy pH (self-calibrating) S/N 1228295055	2008
	Denver Instrument (spare)	UB-5 S/N UB-5093011	2004
Metals ICP	Thermo Jarrell Ash (I-5)	Trace Analyzer 61E, S/N 273490	1994
	Thermo Jarrell Ash (I-6)	Trace Analyzer 61E, S/N 269490	1994
	Thermo ICAP 6500 Duo Ash (I-9)	Trace Analyzer, S/N ICP 20102403	2010
Metals ICP/MS	Thermo (I-8)	Series 2, S/N 01137C	2007

Instrument Type	Manufacturer	Model/Serial No.	Year into Service
Metals Mercury Analyzer	Leeman (CVAA) (H1)	PS200 II, S/N HG9031	1999
	Leeman (CVAA) (H4)	Hydra AA , S/N 6011	2006
WC Autotitrator	Man-Tech	PC – Titrate, S/N MS-9K8-217	2001
WC Block Digester	Andrews	2210 Phenol	1999
	Andrews	2205 Ammonia	1999
	Lachat	BD46 TKN	1992
	Lachat	BD-46 TKN, S/N 00000993	2010
WC BOD	Mantech	BOD, S/N N817947	1999
WC Conductivity	ManTech	4310, S/N 1613	1989
WC Cyanide	LabCrest MidiDist	PRG-2520-BL, S/N 1000-99-01	1999
WC Discrete Analyzer	Kone	Konelab 200, Z1718383	2001
	Kone	Konelab 250, A2120021	2005
WC Dissolved Oxygen Meter	YSI	52C E, S/N 99C1094	1993
WC Flashpoint	Herzog	HFP 339, S/N 073390084	2007
WC Ion Chromatograph	Dionex	DX-320, S/N 00060187	2001
	Dionex	DX-120, S/N 98110093	1999
WC pH Meter	Orion pH Meter	320, S/N 020032	2007
	Orion (Ammonia ISE)	520A, S/N 48029	1996
WC Residual Chlorine Meter			
	Hanna	HI 93701	2006
WC TOC	OI Analytical	1010 TOC Analyzer, S/N K503710931	2005
WC EOX			
	Thermo Electron	1200, S/N 2005.0234	2005

Instrument Type	Manufacturer	Model/Serial No.	Year into Service
WC Turbidimeter	HF Scientific	Micro 100, S/N 200705143	2001
WC UV / VIS			
	Genesys	Spectronic 20, S/N 3SGL078016	1998
	Genesys	Spectronic 20, S/N 3SGL226006 (Model 4001/4)	2008
WC Sulfide	Westco EasyDist		2008
Specialty Analysis-Low Level Mercury	Leeman (CVAF) Low Level (H3)	Hydra AF Gold+, Model #112-00067-1 S/N AFG1006	2001
	Leeman (CVAA) (H4)	Hydra AA , S/N 6011	2006
Specialty Analysis Arsenic Speciation	Trace Detect	Nano-Band Explorer, S/N NBE00017	2003
Specialty Analysis – RSK-175	Agilent (Z)	6890 EPC & PDD/FID, S/N 10205072	2000
Specialty Analysis – Methyl Mercury	Agilent (N)	7890 Atomic Fluorescence, S/N CN10820009 (MeHg)	2008
Specialty Analysis - Autosampler	Agilent (Z)	7694	2000
	EST (N)	Centurion (MeHg)	2008
Specialty Analysis – Purge and Trap	Tekmar (N)	Stratum (MeHg)	2008
	PS Analytical	Model 10.750 (MeHg)	2008

Table 20-2. Schedule of Routine Maintenance
(Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations. Refer to the analytical SOP for frequency and criteria)

INSTRUMENT MAINTENANCE SCHEDULE
ION CHROMATOGRAPH

As Needed	Daily	Weekly	Monthly
Clean micro-membrane suppressor when decreases in sensitivity are observed.	Check plumbing/leaks	Check pump heads for leaks	Check all air and liquid lines for discoloration and crimping, if indicated.
Check fuses when power problems occur.	Check gases	Check filter (inlet)	Check/change bed supports guard and analytical columns, if indicated.
Reactivate or change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated.	Check pump pressure		
De-gas pump head when flow is erratic.	Check conductivity meter		

INSTRUMENT MAINTENANCE SCHEDULE
HIGH PRESSURE LIQUID CHROMATOGRAPH

Daily	**As Needed**
Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse gas and delivery lines to prevent contamination of the new solvent.	Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements.
Check gas supply.	Oil autosampler slides when sample does not advance.
Flush with an appropriate solvent to remove all bubbles.	Rinse flow cell with 1N nitric acid if sensitivity low.
Pre-filter all samples.	Change pump seals when flow becomes inconsistent.
	Repack front end of column Backflush column.

INSTRUMENT MAINTENANCE SCHEDULE
ICP AND ICP/MS

Daily	Monthly or As Needed	Semi-Annually	Annually
Check vacuum pump gage. (<10 millitorr)	Clean plasma torch assembly to remove accumulated deposits	Change vacuum pump oil	Notify manufacturer service engineer for scheduled preventive maintenance service
Check cooling water supply system is full and drain bottle is not full. Also drain tubing is clear, tight fitting, and has few bends.	Clean nebulizer and drain chamber; keep free flowing to maintain optimum performance	Replace coolant water filter (may require more or less frequently depending on quality of water)	
Check nebulizer is not clogged	Clean filters on back of power unit to remove dust		
Check capillary tubing is clean and in good condition	Replace when needed: - peristaltic pump tubing - sample capillary tubing - autosampler sipper probe		
Check peristaltic pump windings are secure	- Check yttrium position - Check O-rings - Clean/lubricate pump rollers		
Check high voltage switch is on			
Check torch, glassware, aerosol injector tube, and bonnet are clean			

INSTRUMENT MAINTENANCE SCHEDULE
CVAS AND CVAFS

Daily	As Needed	Annually
Change drying tube	Change pump tubing	Change Hg lamp
Check pump tubing/drain tubing	Check/change Hg lamp	
Check gas pressure	Clean optical cell	
Check aperture reading Check tubing	Lubricate pump	

INSTRUMENT MAINTENANCE SCHEDULE
GAS CHROMATOGRAPH

Daily *	**As Needed**
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or break off front portion of capillary columns. Replace column if this fails to restore column performance, or when column performance (e.g., peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Quarterly FID: clean detector, only as needed ~~–~~ not quarterly/or semi-annually.
Check temperatures of injectors and detectors. Verify temperature programs by RT shift.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.
Clean injector port weekly for TPH for 8015B, when breakdown fails; otherwise, when RT shift or bad samples run.	Annually FID: replace flame tip, only as needed. Only as needed: ECD--detector cleaning and re-foiling, whenever loss of sensitivity, erratic response, or failing resolution is observed
Check baseline level during analysis of run ~~–~~ not maintenance.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).
Watched weekly: check reactor temperature of electrolytic conductivity detector. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks, when analyzing pesticides; part of analysis ~~–~~ not maintenance. Clip column leader when chromatography looks bad ~~–~~ not daily.	Replace or repair flow controller if constant gas flow cannot be maintained. Replace fuse. Reactivate external carrier gas dryers. Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace ignitor. ECD: follow manufacturer's suggested maintenance schedule. Reactivate flow controller filter dryers when presence of moisture is suspected. HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents.

*No daily maintenance done on any instrument/method. Weekly change IPL on TPH instrument. Everything else is on an "as needed" basis.

INSTRUMENT MAINTENANCE SCHEDULE
MASS SPECTROMETER

Daily	Weekly As Needed		Quarterly	Annually
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.	Check mass calibration (PFTBA or FC-43)	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between maintenance.	Check ion source and analyzer (clean, replace parts as needed)	Replace the exhaust filters on the mechanical rough pump every 1-2 years.
Check temperatures of injector, detector. Verify temperature programs.		Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.	Check vacuum, relays, gas pressures and flows	
Check inlets, septa		Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.	Change oil in the mechanical rough pump.	
Check baseline level		Repair/replace jet separator.		
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		Replace filaments when both filaments burn out or performance indicates need for replacement.		

INSTRUMENT MAINTENANCE SCHEDULE
ANALYTICAL/TOP LOADING BALANCES

Daily	Annually
Check using Class 1-verified weights once daily or before use Clean pan and weighing compartment	Manufacturer cleaning and calibration

INSTRUMENT MAINTENANCE SCHEDULE
REFRIGERATORS/WALK-IN COOLERS

Daily	As Needed
Temperatures checked and logged	Refrigerant system and electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
OVENS

Daily	As Needed
Temperatures checked and logged	Electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
SPECIFIC DIGITAL ION ANALYZER

Daily	As Needed
Daily when used: • Calibrate with check standards • Inspect electrode daily, clean as needed • Inspect electrode proper levels of filling solutions daily; fill as needed • Clean probe after each use	Electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
TURBIDIMETER

Daily	Monthly	As Needed
Daily when used: • Adjust linearity on varying levels of NTU standards. Standardize with NTU standards • Inspect cells	Clean instrument housing	Electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
DISSOLVED OXYGEN METER

Daily	As Needed
Daily when used: • Calibrate with saturated air • Check probe membrane for deterioration • Clean and replace membrane with HCl solution	• Electronics serviced • Clean and replace membrane with HCl solution

INSTRUMENT MAINTENANCE SCHEDULE
CONDUCTANCE METER

Daily	As Needed
Daily when used: • Check probe and cables • Inspect conductivity cell	Electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
CHEMICAL OXYGEN DEMAND (COD) REACTOR [1]

Daily	As Needed
Daily when used: • Calibrate with check standards	Electronics serviced

INSTRUMENT MAINTENANCE SCHEDULE
SPECTROPHOTOMETER

As Needed	Daily	Monthly	Annually
Dust the lamp and front of the front lens	Check the zero % adjustment	Clean windows	Check instrument manual
	Clean sample compartment		Perform wavelength calibration
	Clean cuvettes		Replace lamp annually or when erratic response is observed
			Clean and align optics

INSTRUMENT MAINTENANCE SCHEDULE
pH METER

As Needed	Daily
Clean electrode	Inspect electrode. Verify electrodes are properly connected and filled
Refill reference electrode	Inspect electrode proper levels of filling solutions. Make sure electrode is stored in buffer

INSTRUMENT MAINTENANCE SCHEDULE
TOTAL ORGANIC CARBON ANALYZER

Daily	As Needed	Weekly	Monthly
Check: • Oxygen supply • Persulfate supply • Acid supply • Carrier gas flow rate (~ 150 cc/min) • IR millivolts for stability (after 30 min. warm-up) • Reagent reservoirs	Check injection port septum after 50-200 runs Tube end-fitting connections after 100 hours or use Indicating drying tube NDIR zero, after 100 hours of use Sample pump, after 2000 hours for use Digestion vessel/condensation chamber, after 2000 hours of use Permeation tube, after 2000 hours of use NDIR cell, after 2000 hours of use Change pump tubing	Check liquid-flow-rate-pump-tubing conditions on autosampler Check injection port septum	Clean digestion vessel Clean condenser column Do the leak test

Instrument Maintenance Schedule
Digestion Block

Annually
Check temperature with NIST thermometer

Instrument Maintenance Schedule
Flash Point Tester

Daily
Check tubing Clean sample cup each use
Check gas
Clean flash assembly
Check stirrer

Table 20-3. Preventive Maintenance Procedures
(Note: Refer to the analytical SOP for frequency and criteria.)

SUMMARY OF INORGANIC METHOD CALIBRATIONS

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Acidity	Initial	350.1	Two-level calibration that bracket the expected pH of the sample ± 0.05 pH units of true value		
	Continuing	350.1	One buffer check every 10 samples ± 0.05 pH units		
	Other	350.1	Third point check		
	Ending	350.1	One buffer check ± 0.05 pH units		
Alkalinity, Bicarbonate, Carbonate	Initial	310.1 2320B	2 point calibration of pH meter ± 0.05 pH units of true value	--	N/A
	Continuing	310.1 2320B	One buffer check ± 0.05 pH units of true value Everyone 10 samples	--	N/A
	Ending	310.1 2320B	N/A	--	N/A
Ammonia	Initial	350.1	6 levels including blank, "r" [3] ≥ 0.995	--	N/A
	Continuing	350.1	One level or LCS every 10 samples ± 10% of true value	--	N/A
	Ending	350.1	One level or LCS every 10 samples ± 10% of true value	--	N/A
Arsenic Speciation		N/A	N/A	7063	* Refer to Section 10 of SOP NC-WC-0090

Analysis	Calibration	NPDES [1] Method	NPDES [1] Requirement	RCRA (SW846) [2] Method	RCRA (SW846) [2] Requirement
Biochemical Oxygen Demand (BOD)	Initial	405.1 SM5210B	a. Winkler titration: Iodometric with standard thiosulfate b. Membrane electrode: Read in air and in water with zero dissolved oxygen	--	N/A
	Continuing	405.1 SM5210B	N/A	--	N/A
	Ending	405.1 SM5210B	N/A	--	N/A
Anions, Bromide, Chloride, Fluoride, Sulfate, Nitrite, Nitrate, o-Phosphate	Initial	300.0A	5 levels plus a blank, "r"[3] ≥ 0.995	9056A	5 levels plus a blank, "r" [3] ≥ 0.995
	Continuing	300.0A	Level every 10 samples ± 10% of true value	9056A	N/A
	Ending	300.0A	N/A	9056A	N/A
Chemical Oxygen Demand (COD)	Initial	410.4 SM5220D	5 levels plus a blank"r"[3] ≥ 0.995	--	N/A
	Continuing	410.4 SM5220D	One level every 10 samples ± 10% of true value	--	N/A
	Ending	410.4 SM5220D	One level ± 10% of true value	--	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Chloride	Initial	325.2 SM4500 Cl-E	5 levels plus blank "r" [3] ≥ 0.995	9251	5 levels plus blank "r" [3] ≥ 0.995
	Continuing	325.2 SM4500 Cl-E	One level every 10 samples ± 10% of true value	9251	One level every 10 samples, ± 10% of true value
	Ending	325.2 SM4500 Cl-E	One level every 10 samples ± 10% of true value	9251	Method 9056: N/A Method 9252: One level ± 10% of true value
Chromium Cr^{+6}	Initial	3500 Cr-D 3 levels plus blank		7196A	5 levels plus blank "r" [3] ≥ 0.995
	Continuing	3500 Cr-D	One level every 10 samples ± 10% of true value	7196A	One level every 10 samples ± 15%
	Ending	3500 Cr-D	One level ± 10% of true value	7196A	One level ± 15%
Chlorine, Residual	Initial	330.5 SM4500CL-G	N/A	--	N/A
	Continuing	330.5 SM4500CL-G	N/A	--	N/A
	Ending	330.5 SM4500CL-G	N/A	--	N/A

Analysis	Calibration	NPDES [1] Method	NPDES [1] Requirement	RCRA (SW846) [2] Method	RCRA (SW846) [2] Requirement
Conductivity	Initial	120.1 SM2510B	Standard KCl solution	9050A	One level to determine cell constant
	Continuing	120.1 SM2510B	N/A	9050A	N/A
	Ending	120.1 SM2510B	N/A	9050A	N/A
Cyanide (Amenable)	Initial	335.1 SM4500CN-G	6 levels plus blank "r" [3] ≥ 0.995	9012A	6 levels plus blank "r" [3] ≥ 0.995
	Continuing	335.1 SM4500CN-G	One level every 10 samples ± 10% of true	9012A	One mid-level every 10 samples ± 15% of true value
	Ending	335.1 SM4500CN-G	One level ± 10 % of true value	9012A	± 15% of true value
Cyanide (Total)	Initial	335.2 335.4 SM4500CN-E 335.2-CLP-M (Ohio VAP)	6 levels plus blank "r" [3] ≥ 0.995	9012A	6 levels plus blank "r" [3] ≥ 0.995
	Continuing	335.2 335.4 SM4500CN-E 335.2-CLP-M (Ohio VAP)	One mid-level every 10 samples ± 10 % of true value	9012A	One mid-level every 10 samples ± 15% of true value
	Ending	335.2 335.4 SM4500CN-E 335.2-CLP-M (Ohio VAP)	One mid-level ± 10 % of true value	9012A ±	15% of true value
Cyanide (Weak Acid Dissociable)	Initial	SM 4500 CN-I	6 levels plus blank "r" [3] ≥ 0.995		
	Continuing	SM 4500 CN-I	One mid-level every 10 samples ± 10 % of true value		
	Ending	SM 4500 CN-I	One mid-level ± 10 % of true value		

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Flashpoint	Initial	--	N/A	1010	p-Xylene reference standard must have flashpoint of 81°F ±2°F
	Continuing	--	N/A	1010	N/A
	Ending	--	N/A	1010	N/A

Analysis	Calibration	NPDES [1] Method Requirement	NPDES [1] Method Requirement	RCRA (SW846) [2]	RCRA (SW846) [2]
Fluoride	Initial	340.2 SM 4500 F-C	5 levels "r" [3] ≥ 0.995		
	Continuing	340.2 SM 4500 F-C	One mid-level every 10 samples ± 10% of true value		
	Ending	340.2 SM 4500 F-C	One mid-level ± 10% of true value		
Hardness	Initial	130.2 SM 2340B SM2340C	Method 130.2: Standardize titrant Method 2340B: See ICP Metals 200.7	--	N/A
	Continuing	130.2 SM2340B SM2340C	Method 130.2: N/A Method 2340B: See ICP Metals 200.7	--	N/A
	Ending	130.2 SM2340B SM2340C	Method 130.2: N/A Method 2340B: See ICP Metals 200.7	--	N/A
Iron (Ferrous)	Initial	SM3500-Fe D	3 levels plus a blank, "r" [3] ≥ 0.995	-	N/A
	Continuing	SM3500-Fe D	One mid-level every 10 samples ± 10% of true value	-	N/A
	Ending	SM3500-Fe D	One mid-level ± 10% of true value	-	N/A

Analysis	Calibration	NPDES [1] Method	NPDES [1] Requirement	RCRA (SW846) [2] Method	RCRA (SW846) [2] Requirement
Phosphorus (Total and Ortho-phosphate)	Initial	365.1 SM4500P-E	5 levels plus a blank	--	N/A
	Continuing	365.1 SM4500P-E	One level for every 10 samples. ±10% of true value	--	N/A
	Ending	365.1 SM4500P-E	±10% of true value	--	N/A
pH	Initial	150.1 SM4500H-B	2 level calibration that bracket the expected pH of the sample ± 0.05 pH units of true value	9040B 9040C 9041A 9045C	2 point calibration ± 0.05 pH units of true value
	Continuing	150.1 SM4500H-B	One buffer check every 10 samples ± 0.05 pH units true value	9040B 9040C 9041A 9045C	N/A
	Other	150.1 SM4500H-B	Third point check	9040B 9040C 9041A 9045C	Third point check
	Ending	150.1 SM4500H-B	One buffer check ± 0.05 pH units of true value	9040B 9040C 9041A 9045C	N/A

Analysis	Calibration	NPDES [1] Method	NPDES [1] Requirement	RCRA (SW846) [2] Method	RCRA (SW846) [2] Requirement
Phenolics	Initial	420.1	5 levels plus a blank "r" [3] ≥ 0.995	9065	5 levels plus a blank "r" [3] 0.995
	Continuing	420.1	One mid-level every 10 samples ± 10% true value	9065	One mid-level ± 10% true value
	Ending	420.1	One mid-level ± 10% true value	9065	One mid-level ± 10% true value
Settleable Solids	Initial	160.5 SM2540F	N/A	--	N/A
	Continuing	160.5 SM2540F	N/A	--	N/A
	Ending	160.5 SM2540F	N/A	--	N/A
Sulfate	Initial	375.4	Method 375.4: 3 levels plus blank "r" [3] ≥ 0.995	9038	3 levels plus a blank for every hour of continuous sample analysis.

Analysis	Calibration	NPDES[1] Method	NPDES[1] Requirement	RCRA (SW846)[2] Method	RCRA (SW846)[2] Requirement
Sulfate (Cont'd)	Continuing	375.4	One level every 3 or 4 samples ± 10% of true value	9038	Independent-prepared check standard every 15 samples
	Ending	375.4	± 10% of true value	9038	N/A
Sulfide	Initial	376.1 SM4500S 2-E	This is a titration method. Therefore, calibrations are not applicable.	9030B/ 9034	This is a colorimetric titration. Therefore, calibration is not applicable.
	Continuing	376.1 SM4500S 2-E	N/A	9030B/ 9034	This is a colorimetric titration. Therefore, calibration is not applicable.
	Ending	376.1 SM4500S 2-E	N/A	9030B/ 9034	This is a colorimetric titration. Therefore, calibration is not applicable.
Total Dissolved Solids	Initial	160.1 SM2540C	This is a gravimetric determination. Calibrate balance prior to analysis	--	N/A
	Continuing	160.1 SM2540C		--	N/A
	Ending	160.1 SM2540C		--	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Total Kjeldahl Nitrogen (TKN)	Initial	351.3 SM4500Norg C	Method 351.3: Titrimetric: Standardize titrant Colorimetric: 7 levels plus blank	--	N/A
	Continuing	351.3 SM4500Norg C	Method 351.3: N/A	--	N/A
	Ending	351.3 SM4500Norg C	Method 351.3: N/A	--	N/A
Total Organic Carbon (TOC)	Initial	415.1 SM5310C	3 levels plus blank	9060 Walkley Black	3 levels plus blank "r" [3] $\geq$ 0.995
	Continuing	415.1 SM5310C	1 mid-level every 10 samples ± 10% of true value	9060 Walkley Black	1 mid-level every 10 samples ± 15% of true value
	Ending	415.1 SM5310C	± 10% of true value	9060 Walkley Black	± 15% of true value
Total Organic Halides (TOX)	Initial			(EOX)	Daily instrument calibration standard and blank in duplicate ± 10% of true value (calibration standard) Verify with independently-prepared check standard –ICV ± 10%

Analysis	Calibration	NPDES[1] Method	NPDES[1] Requirement	RCRA (SW846)[2] Method	RCRA (SW846)[2] Requirement
Total Organic Halides (TOX) (cont'd)	Continuing			9023 (EOX)	CCV ± 10% of true value
	Ending			9023 (EOX)	CCV ± 10% of true value
Total Solids	Initial	160.3	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.3		--	N/A
	Ending	160.3		--	N/A
Total Suspended Solids (Nonfilterable)	Initial	160.2 SM2540D	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.2 SM2540D		--	N/A
	Ending	160.2 SM2540D		--	N/A
Turbidity	Initial	180.1	Minimum of 1 level in each instrument range. Follow manufacturer's instructions	--	N/A
	Continuing	180.1	± 10% of true value	--	N/A
	Ending	180.1	± 10% of true value	--	N/A
Volatile Solids	Initial	160.4	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.4		--	N/A
	Ending	160.4		--	N/A

Analysis	Calibration	NPDES[1] Method	NPDES[1] Requirement	RCRA (SW846)[2] Method	RCRA (SW846)[2] Requirement
ICP & ICP/MS Metals (excludes Hg)	Initial	200.7	One level and blank. ICV RSD <3% from replicate - daily	6010B 6010C	One level and blank. ICV RSD <5% from replicate - daily
	Initial	200.8	One level and blank	6020 6020A	One level and blank
	Continuing	200.7	Every 10 samples ±10% of true value CCV RSD < 5% from replicate	6010B 6010C	Mid-level calibration standard Every 10 samples ± 10% of true value CCV RSD < 5% from replicate
	Continuing	200.8	N/A	6020 6020A	N/A
	Ending	200.7	±10% of true value CCV RSD < 5% from replicate	6010B 6010C	Mid-level calibration standard ± 10% of true value CCV RSD < 5% from replicate
	Ending	200.8	N/A	6020 6020A	N/A
	Other	200.7	ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see SOP Semi-Annually: ICP interelement correction factors Instrument detection limits	6010B 6010C	ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see SOP Semi-Annually: ICP interelement correction factors Instrument detection limits
	Other	200.8	N/A	6020 6020A	N/A

Analysis	Calibration	NPDES[1] Method	NPDES[1] Requirement	RCRA (SW846)[2] Method	RCRA (SW846)[2] Requirement
Mercury by CVAA & CVAFS	Initial	245.1 1631E	5 levels plus blank ICV ±10% of true value "r"[3] ≥ 0.995	7470A 7471A 7471B	5 levels plus blank ICV ± 10% of true value "r"[3] ≥ 0.995
	Continuing	245.1* 1631E	Daily or every 10 samples, whichever is more frequent ±20% of true value	7470A 7471A 7471B	Every 10 samples ±20% of true value
	Ending	245.1 1631E	±20% of true value	7470A 7471A 7471B	±20% of original prepared standard
	Other	245.1 1631E	Annually: MDL	7470A 7471A 7471B	Annually: MDL

* 245.1 continuing – Initial CCV±5% of true value

Footnotes

[1] National Pollutant Discharge Elimination System.

[2] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December, 1996).

[3] "r" = correlation coefficient.

SUMMARY OF ORGANIC METHOD CALIBRATIONS

Analysis	Calibration	NPDES[1] Method	NPDES[1] Requirement	RCRA (SW846)[2] Method	RCRA (SW846)[2] Requirement
Herbicides by GC	Initial			8151A	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing			8151A	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending			8151A	Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported.

Analysis	Calibration Method	NPDES [1] Requirement	NPDES [1] Method	RCRA (SW846) [2]	RCRA (SW846) [2] Requirement
Pesticides/ PCBs by GC	Initial	608	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8081A 8081B 8082 8082A	Minimum of 5 levels. If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. (See SOP NC-GC-038)
	Continuing	608	One or more calibration standards analyzed daily. % D ± 15% of predicted response	8081A 8081B 8082 8082A	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	608	N/A	8081A 8081B 8082 8082A	Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported.
	Other	608	N/A	8081A 8081B 8082 8082A	N/A
Petroleum Hydrocarbons/ Oil and Grease	Initial	1664A	Calibrate analytical balance at 2 mg and 1000 mg Calibration must be± 10% at 2 mg and± 0.5% at 1000 mg or recalibrate balance	9071B	Calibrate analytical balance at 2 mg and 1000 mg Calibration must be± 10% at 2 mg and± 0.5% at 1000 mg or recalibrate balance
	Continuing	1664A	N/A	9071B	N/A
	Ending	1664A	N/A	9071B	N/A

Analysis	Calibration	NPDES [1]		RCRA (SW846) [2]	
		Method	Requirement	Method	Requirement
Semivolatiles	Initial	625	Minimum of 3 levels, lowest near but above MDL. If % RSD ≤ 35%, use avg RF. Otherwise calibration curve employed.	8270C 8270D	Minimum of 5 levels, % RSD for RF for CCCs[4] < 30% SPCCs[5]: RF > 0.050
	Continuing	625	One level every 24 ours. Acceptance criteria are found in the method and SOP.	8270C 8270D	Mid-level standard every 12 hours (after tuning) %D for CCCs[4] < 20 % between RF from standard and avg RF from initial SPCCs[5]: RF > 0.050.
	Ending	625 N/A		8270C 8270D	N/A
	Other	625	DFTPP[7] tuning every 24 hours before standard or sample runs.	8270C 8270D	DFTPP[7] tuning at the beginning of every 12 hour shift.

Analysis	Calibration	NPDES [1] Method	NPDES [1] Requirement	RCRA (SW846) [2] Method	RCRA (SW846) [2] Requirement
Volatiles	Initial	624	Minimum of 3 levels, lowest near but above MDL. If % RSD ≤ 35%, use avg RF. Otherwise, calibration curve employed.	8260B 8260C	Minimum of 5 levels, %RSD for RF for CCCs[4] < 30.0% SPCCs[5]:RF ≥ 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1- dichloroethane, and RF > 0.100 for Bromoform
	Continuing	624	1 level every 24 hours Acceptance criteria are found in the method and SOP	8260B 8260C	Mid-level standard every 12 hours (after tuning) %Drift for CCCs(4) < 20.0% between RF from standard and avg RF from initial SPCCs(5): RF ≥ 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1-dichloroethane, and RF > 0.100 for Bromoform
	Ending	624	N/A	8260B 8260C	N/A
	Other	624	BFB(6) tuning at the beginning of every 24 hour shift.	8260B 8260C	BFB(6) tuning at the beginning of every 12 hour shift.

Footnotes:

[1] National Pollutant Discharge Elimination System.

[2] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

[3] TCDD - 2,3,7,8-Tetrachlorodibenzo-p-dioxin.

[4] CCC - Continuing Calibration Compounds.

[5] SPCC - System Performance Check Compound.

[6] BFB – Bromofluorobenzene.

[7] DFTPP – Decafluorotriphenylphosphine.

[8] Footnote deleted.

[9] Method not listed in 40 CFR Part 136.

SECTION 21

MEASUREMENT TRACEABILITY

21.1 OVERVIEW

Traceability of measurements must be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard must be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices (refer to Section 20.3). With the exception of Class A glassware and glass microliter syringes , quarterly accuracy checks are performed for all mechanical volumetric devices. Microsyringes are verified at least semi-annually or disposed of after six months of use. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A glassware and glass microliter syringes should be routinely inspected for chips, acid etching, or deformity (e.g., bent needle). If the Class A glassware or syringe are suspect, the accuracy of the glassware must be assessed prior to use. Actions to correct or segregate ancillary equipment that does not meet required specifications are identified in the calibration and maintenance section of SOPs and maintenance logbooks for the specific equipment.

21.2 NIST-TRACEABLE WEIGHTS AND THERMOMETERS

Reference standards of measurement must be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3 REFERENCE STANDARDS / MATERIALS

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP, with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of

Analysis, the purity of the standard is documented b y analysis. (Refer to Section 9 for additional information on purchasing). The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor-certified different lot is acceptable for use as a second source. For unique situations, where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g., calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to The Corporate Environmental Health & Safety Manual (CW-E-M-001) or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference matearials must not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Qaulity Assurance Manager. The laboratory must have documented contingency procedures for reverifying expired standards. Some regulatory programs, such as Ohio VAP, prohibit the use of reverified standards.

21.4 DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND REFERENCE MATERIALS

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. Refer to TestAmerica's Corporate SOP CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

All manufacturer or vendor-supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in each group. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the

assay purity is less than 96%, a correction must be made to concentrations applied to solutions prepared from the stock commercial material.

21.1.1 21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system , and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation date
- Expiration date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent standard analyte concentration (if applicable)
- Parent standard amount used (if applicable)
- Component analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically in each group for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date, and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration date (include prep date for reagents)
- Standard ID (from LIMS)
- Special health/safety warnings, if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date or preparation for laboratory prepared items. Special health/safety warnings must also be available to the analyst. This information is maintained in the analytical SOP.

21.4.3 In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended storage conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date, and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority:

1) With the manufacturer's recommendations
2) With requirements in the specific analytical methods as specified in the laboratory SOP

SECTION 22

SAMPLING

22.1 OVERVIEW

The laboratory provides sampling services. Sampling procedures are described in SOP NC-SC-006, Sample Procurement Protocol.

22.2 SAMPLING CONTAINERS

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are available from the vendor electronically, or stored at the laboratory.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the Chain-of-Custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time ends 24 hours after sampling. Holding times for analysis include any necessary re-analysis. However, there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of holding time length.

22.4 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS , HOLDING TIMES

The preservation and holding time criteria specified in the following tables are derived from the source documents for the methods. If method-required holding times (refer to Tables 23-1 to 23-7 and in SOPs) or preservation requirements are not met, the reports must be qualified using a flag, footnote, or case narrative. As soon as possible, or "ASAP", is an EPA designation for tests for which rapid analysis is advised; but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 SAMPLE ALIQUOTS / SUBSAMPLING

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample needs consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative sub-sample or aliquot of the sample provided for analysis. In that regard the following guidelines apply to analysts:

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots and sub-sampling are located in each analytical SOP.

Tables 23-1 to 23-7 detail holding times, preservation and container requirements, and sample volumes for NPDES methods . The sample volumes are intended to be a minimal amount to perform the method. The containers used may be of larger size.

Please note: The holding times are program specific and different programs may have different holding times for equivalent methods, e.g., there are differences in holding times for many organic analytes between RCRA and NPDES.

Table 22-1. Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Acidity	Water	100 mL	305.1 SM2310B	250 mL plastic or glass. Cool to 4°C, 14 days	---	N/A
Alkalinity, Bicarbonate, Carbonate	Water	100 mL	310.1 SM2320B	250 mL plastic or glass. Cool to 4°C, 14 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Ammonia	Water	100 mL	350.2 SM4500NH_3-E SM4500NH_3-B	500 mL plastic or glass. Cool to 4°CH_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Arsenic (ASV) Anodic Stripping Voltammetry	Water	100 mL	---	N/A	7063	250 mL plastic. Cool to 4°C. HCl to pH <2, 28 days
Biochemical Oxygen Demand (BOD), Carbonaceous	Water	1000 mL	405.1 SM5210B	1000 mL plastic or glass. Cool to 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Anions, Bromide, Chloride, Fluoride, Sulfate,	Water	50 mL	300.0A [7]	250 mL plastic or glass. No preservative required, 28 days	9056A	Cool to 4°C. Analyze ASAP after collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Anions, Nitrate, Nitrite, ortho-Phosphate	Water	50 mL	300.0A [7]	250 mL plastic or glass. Cool to 4°C, 48 hours.	9056A	Cool to 4°C. Analyze within 48 hours of collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Chemical Oxygen Demand	Water	100 mL	410.4 5220D	250 mL glass or plastic. Cool to 4°C, H_2SO_4 to pH < 2,	---	N/A

Analytical Parameters (COD)	**Matrix**	**Minimum Sample Size** [1]	**NPDES** [2, 3, 7]		**RCRA (SW846)** [3, 4]	
			Method	**Requirements** 28 days	**Method**	**Requirements**
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Chloride	Water	50 mL	325.2 SM 4500-Cl-E	250 mL plastic or glass. No preservative required, 28 days	9251	Method 9251: 250ml plastic or glass, no preservative required, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Chlorine, Residual	Water	100 mL	330.5 SM 4500 Cl-G	250 mL glass or plastic. Cool to 4°C, analyze immediately	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Chromium (Cr^{+6})	Water	100 mL	3500 Cr-D	Method 3500 Cr-D: 200 mL quartz, TFE, or polypropylene HNO_3 to pH <2. Cool to 4°C. Analyze ASAP after collection	7196A	200 mL plastic or glass. Cool to 4°C, 24 hours
	Solid	20 g	---	N/A	7196A 3060A	250 mL plastic or glass, 30 days to digestion, 168 hours after digestion
	Waste	N/A	---	N/A	---	N/A
Conductivity	Water	100 mL	120.1 SM2510B	200 mL glass or plastic. Cool to 4°C, 28 days	9050A	200 mL glass or plastic. Cool to 4°C, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Cyanide (Amenable)	Water	250 mL	335.1 SM4500CN-G	1 liter plastic or glass, NaOH to pH >12 Cool to 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours.	9012A	1 liter plastic or glass, NaOH to pH >12 Cool to 4°C, 14 days
	Solid	50g	---	N/A	9012A	Not Specified
	Waste	50g	---	N/A	9012A	Not Specified
Cyanide (Total)	Water	1L	335.2 335.3 335.4 (7) SM4500CN-E 335.2-CLP-M	1 liter plastic or glass, NaOH to pH >12 Cool to 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours.	9012A	1 liter plastic or glass, NaOH to pH >12 Cool to 4°C, 14 days.
	Solid	50g	--	N/A	9012A	8 or 16 oz glass Teflon-lined lids, Cool to 4°C, 14 days
	Waste	50g	--	N/A	9012A	8 or 16 oz glass Teflon-lined lids, Cool to 4°C
Flashpoint (Ignitability)	Liquid	100 mL	---	N/A	1010 ASTM D93-9	No requirements, 250 mL amber glass. Cool to 4°C recommended
	Solid	100 g	--	N/A	---	N/A
	Waste	100 mL	--	N/A	---	N/A
Fluoride	Water	300 mL	340.2 SM 4500 F-C	500 mL plastic. No preservation required, 28 days.		
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Hardness (Total)	Water	50 mL	130.2 SM2340C	250 mL glass or plastic, HNO_3 to pH < 2, 6 months	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Iron (Ferrous)	Water	100 mL	3500-Fe D	1 liter glass or polyethylene containe. **This test should be performed in the field.**	-	N/A
	Solid	N/A	-	N/A	-	N/A
	Waste	N/A	-	N/A	-	N/A
Ortho-phosphate	Water	50 mL	365.1 SM4500P-E	100 mL plastic or glass. Filter on site. Cool to 4°C, 48 hours		
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
pH	Water	50 mL	150.1 SM4500H-B	100 mL plastic or glass. Analyze immediately. **This test should be performed in the field.**	9040B	100 mL plastic or glass. Analyze immediately. **This test should be performed in the field.** [(8)]
	Solid	N/A	---	N/A	9045C	4 oz glass or plastic. Cool to 4°C. Analyze as soon as possible. [8]
	Waste	N/A	---	N/A	9045C	4 oz glass or plastic, Cool to 4°C. Analyze as soon as possible. [8]
Phenolics	Water	100 mL	420.1	500 mL glass, Cool to 4°C, H_2SO_4 to pH < 2, 28 days	9065	1 liter glass recommended, Cool to 4°C, H_2SO_4 to pH < 4, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	9065	Not Specified

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Phosphorus (Total)	Water	100 mL	365.1 SM4500P-E	100 mL plastic or glass, H_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Settleable Solids	Water	1000 mL	160.5 SM2540F	1000 mL plastic or glass. Cool to 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Sulfate (SO_4)	Water	50 mL	375.4	100 mL plastic or glass. Cool to 4°C, 28 days	9038	200 mL plastic or glass, Cool to 4°C, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	100 mL	---	N/A	9038	200 mL plastic or glass. Cool to 4°C, 28 days

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7] Method	NPDES [2, 3, 7] Requirements	RCRA (SW846) [3, 4] Method	RCRA (SW846) [3, 4] Requirements
Sulfide	Water	250 mL	376.1 SM 4500 S2-E	500 mL plastic or glass. Cool to 4°C, Add 2 mL zinc acetate plus NaOH to pH > 9, 7 days	9030A 9030B/ 9034	500 mL plastic, No headspace. Cool to 4°C. Add 4 drops of 2N zinc acetate per 100 mL of sample, adjust the pH to > 9 with 6 N NaOH solution, 7 days
	Solid	50 g	---	N/A	9030A 9030B/ 9034	Cool to 4°C. Fill surface of solid with 2N Zinc acetate until moistened. Store headspace-free
	Waste	50 g	---	N/A	9030A 9030B/ 9034	Cool to 4°C. Fill surface of solid with 2N Zinc acetate until moistened. Store headspace-free
Total Dissolved Solids (Filterable)	Water	100 mL	160.1 SM2540C	250 mL plastic or glass. Cool to 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Total Kjeldahl Nitrogen (TKN)	Water	100 mL	351.3 SM 4500-Norg-B SM 4500 NH3-E	500 mL plastic or glass. Cool to 4°C, H_2SO_4 to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Total Organic Carbon (TOC)	Water	100 mL	415.1 SM5310D	100 mL plastic or glass. Cool to 4°C, H_2SO_4 to pH < 2, 28 days	9060	100 mL glass or 40 mL VOA vials,Cool to 4°C, H_2SO_4 or HCl to pH < 2, 28 days
	Solid	N/A	---	N/A	Walkley-Black	Not Specified Cool to 4°C, 28 days
	Waste	N/A	---	N/A	Walkley-Black	Not Specified Cool to 4°C, 28 days
Extractable Organic Halides (EOX)	Solid	100 mL			9023 (EOX)	500 mL amber glass, Teflon□-lined lid. Cool to 4°Cno headspace, 28 days
Total Solids	Water	100 mL	160.3	250 mL plastic or glass. Cool to 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Total Suspended Solids (Nonfilterable)	Water	100 mL	160.2	250 mL plastic or glass. Cool, 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3, 7]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method	Requirements
Turbidity	Water	50 mL	180.1	250 mL plastic or glass. Cool, 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Volatile Solids	Water	100 mL	160.4 250	mL plastic or glass. Cool, 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Metals (excludes Hg)	Water	100 mL	200 series	1 liter glass or polyethylene container, HNO_3 to pH ≤ 2, 6 months	6010B 6010C 6020 6020A	1 liter glass or polyethylene container, HNO_3 to pH ≤ 2, 6 months
	Solid	200 g	200 series	2, 8, or 16 oz glass or polyethylene container storage at 4 °C	6010B 6010C 6020 6020A	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
	Waste	200 g	200 series	N/A	6010B 6010C 6020 6020A	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
Mercury (CVAA) (CVAFS)	Water	100 mL	245.1 1631E	250 mL glass or polyethylene container, HNO_3 to pH ≤ 2, 28 days	7470A	1 liter glass or polyethylene container, HNO_3 to pH ≤ 2, 28 days
	Solid	200 g	--	2, 8, or 16 oz glass or polyethylene container. Cool to 4°C, 28 days. Not applicable for Method 1631E.	7471A 7471B	8 or 16 oz glass or polyethylene container. Cool to 4°C, 28 days (CORP-MT-0007)
	Waste	200 g	--	N/A	7471A 7471B	8 or 16 oz glass or polyethylene container. Cool, 4°C, 28 days (CORP-MT-0007)

Footnotes

[1] Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.

[2] National Pollutant Discharge Elimination System - MCAWW, March 1983.

[3] Holding times are calculated from date of collection. Holding Times are determined based on date of collection to preparation/analysis.

[4] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA, (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

[5] Solid matrix type includes soil, sediment, sludge and other solid materials not classified as waste.

[6] Samples to be analyzed for cyanide should be field-tested for residual chlorine. If residual chlorine is detected, ascorbic acid should be added.

[7] Method not listed in 40 CFR Part 136.

[8] If not done in the field (ASAP) per the method and requested by client, analyze in lab within 48 hours.

Table 22-2. Organic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Herbicides	Water	1L			8151A	1 liter amber glass with Teflon□-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool to 4°C. Extraction, 7 days. Analysis, 40 days of the start of extraction.
	Solid	50 g			8151A	4 or 8 oz glass widemouth with Teflon□-lined lid. Cool to 4 °C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.
	Waste	50 g			8151A	4 or 8 oz glass widemouth with Teflon□-lined lid. Cool to 4 °C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Pesticides/ PCBs	Water	1L	608	1 liter amber glass with Teflon□-lined lid, Adjust pH to 5-9 if extraction not to be done within 72 hours of sampling. Add sodium thiosulfate if residual chlorine present and aldrin is being determined. Cool, 4°C. Extraction, 1 year. Analysis, 40 days after extraction.	8081A 8081B 8082 8082A	1 liter amber glass with Teflon□-lined lid, If residual chlorine present, add 3 mL 10% sodium thiosulfate per gallon. Cool, 4°C. Extraction, 7 days (1 year for 8082A). Analysis, 40 days of the start of the extraction.
	Solid	50 g	---	N/A	8081A 8081B 8082 8082A	4 or 8 oz glass wide mouth with Teflon□-lined lid. Cool, 4°C. Extraction, 14 days (1 year for 8082A). Analysis, 40 days of the start of the extraction.
	Waste	50 g	---	N/A	8081A 8081B 8082 8082A	4 or 8 oz glass wide mouth with Teflon□-lined lid. Cool, 4°C. Extraction, 14 days (1 year for 8082A). Analysis, 40 days of the start of the extraction.

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Oil and Grease	Water	1 L	1664A(7)	1 liter glass, Cool, 4°C HCl or H_2SO_4 to pH <2 28 days	9071B	1 liter glass, Cool, 0-4°C HCl or H_2SO_4 to pH <2 28 days
	Solid	30 g	1664A(7)	8 or 16 oz. Wide mouth glass jar, Cool, 4°C, 28 days	9071B	8 or 16 oz. wide mouth glass jar, Cool, 0-4°C, 28 days
	Waste	---	---	N/A	9071B	N/A
Semivolatiles	Water	1L	625	1 liter amber glass with Teflon□-lined lid. Cool, 4°C. Extraction, 7 days. Analysis, 40 days.	8270C 8270D	1 liter amber glass with Teflon□-lined lid, If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C. Extraction, 7 days. Analysis, within 40 days of extraction.
	Solid	50 g	---	N/A	8270C 8270D	8 or 16 oz glass wide mouth with Teflon-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, within 40 days of extraction.
	Waste	50 g	---	N/A	8270C 8270D	8 or 16 oz glass wide mouth with Teflon□-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, within 40 days of extraction.

Analytical Parameters	Matrix	Minimum Sample Size [1]	NPDES [2, 3]		RCRA (SW846) [3, 4]	
			Method	Requirements	Method [6]	Requirements
Volatile Organics	Water	40 mL	624	40 mL glass, VOA vial (in triplicate) with Teflon□-lined septa without headspace. Cool to 4°C. Add sodium thiosulfate if residual chlorine, 7 days with pH > 2, 14 days with pH ≤ 2[8].	8260B 8260C	40 mL glass, VOA vial (in triplicate) with Teflon□-lined septa without headspace. Cool to 4°C. Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days with pH ≤ 2[9].
	Solid[5]	5 g or 25 g	--	N/A	8260B 8260C	4 or 8 oz glass with Teflon□-lined lid. Cool to 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Maximum holding time for EnCore™ sampler is 48 hrs (before the sample is added to methanol or sodium bisulfate). Cool to 4°C[(12)]
	Waste	5 g or 25 g	--	N/A	8260B 8260C	4 or 8 oz glass with Teflon□-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Maximum holding time for Encore™ sampler is 48 hrs (before sample is added to methanol or sodium bisulfate). Cool to 4°C[12]

Footnotes

[1] Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.

[2] National Pollutant Discharge Elimination System - 40 CFR Part 136, Appendix A.

[3] Holding times are calculated from the date of collection.

[4] Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

[5] Solid matrix type includes soil, sediment, sludge or other solids not classified as waste.

[6] Only one determination method is listed when separate methods are required for preparation and analysis.

[7] Method 1664 was promulgated by the EPA with an effective date of June 14, 1999.

[8] For acrolein and acrylonitrile the pH should be adjusted to 4-5. This pH adjustment is not required if acrolein is not measured. Samples requiring analysis of acrolein that received no pH adjustment must be analyzed within three days of sampling.

[9] For acrolein and acrylonitrile the pH should be adjusted to 4-5.

[10] Method not listed in 40 CFR Part 136.

[11] Should only be used in the presence of residual chlorine

[12] Depending on regulatory programs, EnCore™ samplers may be preserved for up to 14 days from sampling by freezing at -5 to-12°C until analysis. Alternatively the EnCore™ sample may be transferred to a 40-ml VOA vial and preserved by freezing at -5 to -12°C until analysis. Some regulatory agencies may require 4 or 8 oz glass with Teflon®-lined lid, Cool 4°C, 14 days. This technique is not recommended, but will be supported where required. (Preservation and holding times are subject to client specifications.)

Table 22-3. Sample Containers, Preservatives, and Holding Times for TCLP[1] and SPLP[2]

Analytical Parameters	Matrix	Minimum Sample Size	TCLP Method 1311 and SPLP Method 1312 Requirements	
			From Field Collection to TCLP/SPLP Extraction	From TCLP/SPLP Extraction to Analysis
Mercury	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 28 days	Glass or polyethylene 28 days
Metals (except mercury)	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 180 days	Glass or polyethylene 180 days
Semivolatiles	Liquid Solid Waste	1L	1L glass, Cool 4°C, 14 days	1L glass Extraction of leachate within 7 days of TCLP extraction, Analyze extract within 40 days
Volatiles	Liquid Solid Waste	6 oz	4 oz glass, Cool 4°C, 14 days	40 mL glass, 14 days

Footnotes

[1] TCLP = Toxicity Characteristic Leaching Procedure
[2] SPLP = Synthetic Precipitation Leaching Procedure

SECTION 23

HANDLING OF SAMPLES

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 CHAIN OF CUSTODY (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the Sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time, and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to TestAmerica personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's Field technician until the samples are delivered to the laboratory personnel. The sample collector must assure each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the Sample Control personnel at the laboratory or to a TestAmerica courier. When sampling personnel deliver the samples through a common carrier (FedEx, UPS), the COC relinquished date/time is completed by the Field personnel; and samples are released to the carrier. Samples are only considered to be received by lab when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The COC is stored with project information and the report.

23.1.2 Legal / Evidentiary Chain-of-Custody

The lab does not accept samples that require legal Chain-of-Custody.

23.2 SAMPLE RECEIPT

Samples are received at the laboratory by designated Sample Receiving personnel, and a unique laboratory project identification number is assigned. Each sample container must be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking, and storage procedures are summarized in the following sections. SOP NC-SC-005, Sample Receiving and Sample Control, describes the laboratory's sample receipt procedure.

23.2.1 Laboratory Receipt

Samples must be received and logged in at TestAmerica by a designated sample custodian or other properly trained associate. Upon sample receipt, the sample custodian shall, as appropriate:

- Wear appropriate personal protective equipment. At a minimum, this consists of cut-resistant gloves, a lab coat, and safety glasses
- Examine the shipping containers to verify that the custody tape is intact
- Examine all sample containers for damage
- Open shipping containers in adequately ventilated areas to assure worker safety
- Determine if the temperature required by the requested testing program has been maintained during shipment. Document the shipping container temperature on the Cooler Receipt Form
- Compare samples received against those listed on the COC
- Verify that sample holding times have not been exceeded
- Examine all shipping records for accuracy and completeness
- Determine sample pH (if required for the scheduled analysis) (except VOA and TOX

samples) and record on the Cooler Receipt Form (CRF)

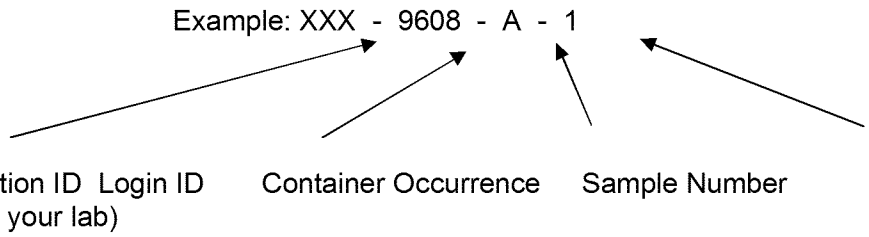
- Sign and date the COC immediately (only after shipment is accepted) and attach the waybill
- Note any problems associated with the coolers and samples on the cooler receipt form and notify the PM who in turn notifies the client
- Attach durable (water-resistant) laboratory sample container labels with unique laboratory identification number and test
- Place the samples in proper laboratory storage.

A Cooler Receipt Form (CRF) or an equivalent form/system is generated by sample control during the sample log-in process to document anomalies identified upon the receipt of samples in the laboratory. These anomalies are outside of laboratory control and do not require corrective actions to be taken within the laboratory. The affected client must be notified by the PM or designee of all issues generated for their samples. The PM is responsible for resolving with the client how to proceed with the samples and documenting the decision to proceed with the analysis of compromised samples. Issues must be resolved prior to sample preparation and analysis. The completed CRF must be stored in the project file. An example CRF is shown in Figure 24-4. The report narrative must include an explanation of sample receiving related anomalies.

23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):



The above example states that TestAmerica <location> Laboratory (Location XXX). Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step th at creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: XXX - 9608 - A - 1 **A** **Secondary Container Occurrence**

Example: 220-9608-A-1-A, would indicate the PRIMARY container listed above that went through a step that created the 1st occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy outlined in SOP NC-SC-005, Sample Receiving and Sample Control, that clearly outlines the circumstances under which samples must be accepted or rejected. These include:

- A COC filled out completely
- Samples must be properly labeled
- Proper sample containers with adequate volume for the analysis and necessary QC
- Samples must be preserved according to the requirements of the requested analytical method
- Sample holding times must be adhered to
- All samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time
- The Project Manager must be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

Once sample acceptance is verified, the samples are logged into LIMS according to SOP NC-SC-005.

23.4 SAMPLE STORAGE

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers, or protected locations suitable for the sample matrix. Metals samples may be unrefrigerated. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards, or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every week.
Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions

of samples, including empty sample containers, are r eturned to the secure sample control area. All samples are kept in the refrigerators for a minimum of 30 days after report generation, which meets or exceeds most sample holding times. After this time period, the samples are removed from the refrigerator shelves and prepared for disposal. Special arrangements may be made to store samples for longer periods of time.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.5 HAZARDOUS SAMPLES AND FOREIGN SOILS

All samples per SOP are treated as hazardous. If any extra or known hazards are present in the sample, the sample is flagged and precautions / instructions are put in the comments. Hazardous samples are segregated out, and go into the waste stream profile for the nature of the hazard. All soils--foreign and domestic--go to a USDA approved incinerator.

23.6 SAMPLE SHIPPING

In the event the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0 °C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses. The Chain-of-Custody form is signed by the Sample Control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper Chain-of-Custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 SAMPLE DISPOSAL

Samples should be retained for a minimum of 30 days after the project report is sent; however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist--the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP NC-SC-005, Sample Receiving and Sample Control). All procedures in the laboratory

Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work. Waste disposal complies with all federal and state laws and regulations.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), and names of individuals who conducted the arrangements and physically completed the task. Sample labels are destroyed through the disposal method, e.g., samples are incinerated. A Waste Manifest is completed.

Figure 23-1. Example: Chain of Custody (COC)

TestAmerica North Canton
4101 Shuffle Drive N.W.
North Canton, OH 44720
phone 330-497-9396 fax 330-497-0772

Chain of Custody Record

Client Contact	**Project Manager:**	**Site Contact:**	**Date:**
Your Company Name here	**Tel/Fax:**	**Lab Contact:**	**Carrier:**
Address	**Analysis Turnaround Time**		
City/State/Zip	Calendar (C) or Work Days (W)		
(xxx) xxx-xxxx Phone	TAT if different from Below _____		
(xxx) xxx-xxxx FAX	☐ 2 weeks		
Project Name:	☐ 1 week		
Site:	☐ 2 days		
P O #	☐ 1 day		

Sample Identification	**Sample Date**	**Sample Time**	**Sample Type**	**Matrix**	**# of Cont.**	**Filtered Sample**

Preservation Used: 1= Ice, 2= HCl; 3= H_2SO_4; 4=HNO_3; 5=NaOH; 6= Other

Possible Hazard Identification
☐ *Non-Hazard* ☐ *Flammable* ☐ *Skin Irritant* ☐ *Poison B* ☐ *Unknown* ☐

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)
☐ *Return To Client* ☐ *Disposal BY Lab* ☐ *Archive For* ☐ *Months*

Special Instructions/QC Requirements & Comments:

Relinquished by:	Company:	Date/Time:	Received by:	Company:
Relinquished by:	Company:	Date/Time:	Received by:	Company:
Relinquished by:	Company:	Date/Time:	Received by:	Company:

Figure 23-2.

Example: Custody Seal

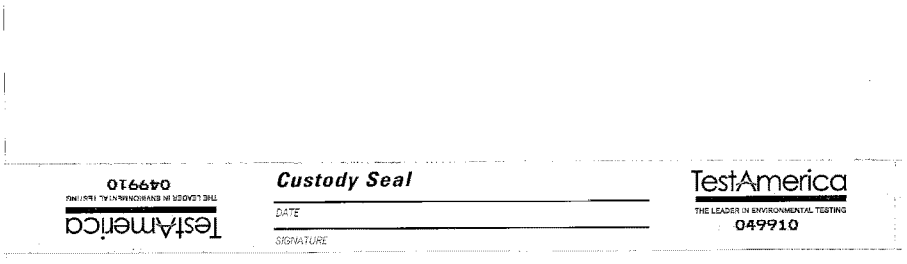


Figure 23-3. Example: Internal Chain of Custody (COC)

TestAmerica Laboratories, Inc.
Sample Control Record

Client:

Lot Number:

Case Number/SDG:

Storage Location:

Laboratory Sample ID	Transferred By	Date	Entered	Removed	Reason	Date Returned

Figure 23-4. Example: Cooler Receipt Form

TestAmerica North Canton Sample Receipt Form/Narrative **Login #** :_____

Client Site Name By:

Cooler Received on Opened (Signature)

FedEx: 1st Grd Exp UPS FAS Stetson Client Drop Off TestAmerica Courier

TestAmerica Cooler # _____ Foam Box Client Cooler Box Other

Packing material used: Bubble Wrap Foam Plastic Bag None Other

COOLANT: Wet Ice Blue Ice Dry Ice Water None

1. Cooler temperature upon receipt
   - IR GUN# 1 (CF -2ºC) Observed Sample Temp._____ºC Corrected Sample Temp._____ºC
   - IR GUN# 4G (CF -1ºC) Observed Sample Temp._____ºC Corrected Sample Temp._____ºC
   - IR GUN# 5G (CF -1ºC) Observed Sample Temp._____ºC Corrected Sample Temp._____ºC
   - IR GUN# 6Y (CF -2ºC) Observed Sample Temp._____ºC Corrected Sample Temp._____ºC
2. Were custody seals on the outside of the cooler(s)? If Yes Yes
   - -Were custody seals on the outside of the cooler(s) signed & dated? Yes NA
   - -Were custody seals on the bottle(s)? Yes
3. Shippers' packing slip attached to the cooler(s)? Yes
4. Did custody papers accompany the sample(s)? Yes
5. Were the custody papers relinquished & signed in the appropriate Yes
6. Did all bottles arrive in good condition (Unbroken)? Yes
7. Could all bottle labels be reconciled with the COC? Yes
8. Were correct bottle(s) used for the test(s) indicated? Yes
9. Sufficient quantity received to perform indicated analyses? Yes
10. Were sample(s) at the correct pH upon receipt? Yes NA
11. Were VOAs on the COC? Yes
12. Were air bubbles >6 mm in any VOA vials? Yes No NA
13. Was a trip blank present in the cooler(s)? Yes

Contacted PM _____ Date _____ by _____ via Verbal Voice Mail

Concerning

14. CHAIN OF CUSTODY & SAMPLE DISCREPANCIES

15. SAMPLE CONDITION

Sample(s)_____were received after the recommended holding time had

Sample(s)_____ were received in a broken

Sample(s)_____were received with bubble >6 mm in diameter.

16. SAMPLE PRESERVATION

Sample(s) _____ were further preserved in Sample Receiving to meet recommended pH level(s). Nitric Acid Lot# 110410-HNO3; Sulfuric Acid Lot# 041911-H2SO4; Sodium Hydroxide Lot# 121809 -NaOH; Hydrochloric Acid Lot# 041911-HCl; Sodium Hydroxide and Zinc Acetate Lot# 100108-(CH3COO)2ZN/NaOH. What time was preservative added to

Client ID	pH	Date	Initials
Cooler #	Observed Sample Temp. ºC Corrected Sample Temp. ºC	IR #	Coolant

Section 24

ASSURING THE QUALITY OF TEST RESULTS

24.1 OVERVIEW

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g., Method Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 CONTROLS

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 NEGATIVE CONTROLS

Table 24-1. Example – Negative Controls

Control Type	Details
Method Blanks (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Re-analyze or quality-associated sample results when the concentration of a targeted analyte in the method blank is at, or above, the reporting limit as established by the method or by regulation, AND is greater than 1/20 of the amount measured in the sample.
Calibration Blanks	are prepared and analyzed along with calibration standards where applicable or injected at specifed frequencie s throughout an analytical sequence. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve. These blanks may be termed Initial Calibration Blanks (ICB) or Continuing Calibration Blanks (CCB),

Table 24-1. Example – Negative Controls

Control Type	Details
Instrument Blanks	are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.
Trip Blanks [1]	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile comp ounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sou rces if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean contain er with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 POSITIVE CONTROLS

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon:

1) Method Performance [Laboratory Control Sample (LCS) or Blank Spike (BS)], which entails both the preparation and measurement steps

2) Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed.

Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch.

Note that frequency of control samples vary with specific regulatory, methodology, and project-specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

24.4.1.1 The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

24.4.1.2 The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

24.4.1.3 Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g., solid matrix LCS for metals, TDS, etc.).

24.4.1.4 The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally one for each batch of sample--not to exceed 20 environmental samples.

24.4.1.5 If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable, e.g., no spike of pH. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

24.4.1.5.1 For methods that have 1-10 target analytes, spike all components.

24.4.1.5.2 For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.

24.4.1.5.3 For methods with more than 20 target analytes, spike at least 16 components.

24.4.1.5.4 Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.

24.4.1.5.5 Exception: Due to analyte incompatibility between the various PCB Aroclors, Aroclors 1016 and 1260 are used for spiking as they cover the range of all of the Aroclors. Specific Aroclors may be used by request on a project-specific basis.

24.5 SAMPLE MATRIX CONTROLS

Table 24-2 Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	To assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried throug h the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects.If the mandated or requested test method does not specify the spiking components, the laborat ory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description Es	sentially, a sample fortified with a known amount of the test analyte(s).
Surrogate Use		Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the control limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2] Use		For a measure of analytical precision, with each matrix- specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD) , or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description Pe	rformed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (inclu ding the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description Us	ed to correct for matri x effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performanc

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.
[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 CONTROL LIMITS

24.6.1 As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project-specific control

limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes, and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Note: For Ohio VAP the laboratory must implement Corrective Action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all corrective actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale will be presented in the final report and/or affidavit and the data will be noted as 'not VAP certified' on the affidavit.

24.6.2 Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

24.6.3 Laboratory-generated Percent Recovery acceptance (control) limits are generally established by taking + _ 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

24.6.3.1 Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV), (unless the analytical method specifies a tighter limit).

24.6.3.2 In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evlauted against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) an be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

24.6.3.3 The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5%, and the analyte must be detectable and identifiable.

24.6.3.4 The maximum acceptable recovery limit will be 200%.

24.6.3.5 The maximum acceptable RPD limit will be 30% for organic methods and 20% for inorganic methods. The minimum RPD limit is 10%.

24.6.3.6 If either the high or low end of the control limit changes by <_ 10% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

24.6.4 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical

control limits. Refer to NC-QA-018, Statistical Eva luation of Data and Development of Control Charts, for details.

24.6.5 An LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the control limits may be determined as out of control and should be re-analyzed if possible. If re-analysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal Corrective Action process (see Section 12) is also initiated if an LCS exceeds the control limits. Sample results may be qualified and reported without re-analysis if:

24.6.5.1 The analyte results are below the reporting limit and the LCS is above the upper control limit.

24.6.5.2 If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

Note: For Ohio VAP the laboratory must implement Corrective Action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all corrective actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale will be presented in the final report and/or affidavit and the data will be noted as 'not VAP certified' on the affidavit.

24.6.5.3 Or, Department Of Defense (DOD) work, there are an allowable number of Marginal Exceedances (ME):

- <11 analytes 0 marginal exceedances are allowed.
- 11 – 30 Analytes 1 marginal exceedance is allowed
- 31-50 Analytes 2 marginal exceedances are allowed
- 51-70 Analytes 3 marginal exceedances are allowed
- 71-90 Analytes 4 marginal exceedances are allowed
- > 90 Analytes 5 marginal exceedances are allowed

24.6.5.3.1 Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit ().

Note: Use of Marginal Exceedances is not permitted for Ohio VAP.

24.6.5.3.2 Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

24.6.5.3.3 Though marginal excedences may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

24.6.6 If the MS/MSDs do not meet control limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and re-analyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.7 If a surrogate standard falls outside the control limits, and if there is not obvious chromatographic matrix interference, re-analyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the re-analysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client).

Note: A more detailed discussion of acceptance criteria and corrective action can be found in the laboratory's method SOPs and in Section 12.

24.7 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

24.7.1 The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21), and use of PT samples (see Section 15).

24.7.2 A discussion regarding MDLs, Limit of Detection (LOD), and Limit of Quantitation (LOQ) can be found in Section 19.

24.7.3 Use of formulae to reduce data is discussed in the method SOPs and in Section 20.

24.7.4 Selection of appropriate reagents and standards is included in Sections 9 and 21.

24.7.5 A discussion on selectivity of the test is included in Section 5.

24.7.6 Constant and consistent test conditions are discussed in Section 18.

24.7.7 The laboratory sample acceptance policy is included in Section 23.

SECTION 25

REPORTING RESULTS

25.1 OVERVIEW

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is a conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory must work with the client during project setup to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

Review of reported data is included in Section 19.

25.2 TEST REPORTS

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed, reviewed, and signed by the appropriate Project Manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title with a "Sample Result" header.

25.2.2 Each report cover page printed, which includes the laboratory name, address, and telephone number.

25.2.3 A unique identification of the report (e.g., Work Order number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented at the bottom of each page. The report is sequentially paginated. The final page of the report is labeled as "End of Report".

25.2.4 A copy of the Chain-of-Custody (COC).

- Any COCs involved with subcontracting are included.

- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (e.g., Sampling information).

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis.

25.2.9 Date reported or date of revision, if applicable

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc)

25.2.11 Reporting limit

25.2.12 Method detection limits (if requested)

25.2.13 Definition of data qualifiers and reporting acronyms, e.g., ND

25.2.14 Sample results

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (refer to Section 25.2.4 – Item 3, regarding additional addenda).

25.2.17 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

25.2.18 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

25.2.18 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

25.2.19 When TNI accreditation is required, the lab must certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.

25.2.20 The laboratory includes a cover page.

25.2.21 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.22 When soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

25.2.23 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.24 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report, e.g., partial report, or how your lab identifies it. A complete report must be sent once all of the work has been completed.

25.2.25 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.26 A clear statement notifying the client that non-accredited tests were performed and directing the client to the laboratory's accreditation certificates of approval shall be provided when non-accredited tests are included in the report.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy CA-L-P-002 for details on internally applying electronic signatures of approval.

25.2.26 Reports for Ohio VAP work require a VAP affidavit be completed and included with the report. One affidavit can be provided for multiple reports for a project.

Note: For additional information on Ohio VAP affidavits refer to OAC Rule 3745-300-04 and OAC RULE 3745-300-13(N), effective March 1, 2009.

25.3 REPORTING LEVEL OR REPORT TYPE

The laboratory offers two levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I is a report with the features described in Section 25.2 above.
- Level II is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.

Level IV is the same as Level III with the addition of all raw supporting data.In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Procedures used to ensure client confidentiality are outlined in Section 25.7.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica services. TestAmerica North Canton offers a variety of EDD formats including (but not limited to) ADR, EQuIS, GISKey, Region 5, NJHAZsite, and a wide variety of client specific multi-file, Excel and flat file formats.

EDD specifications are submitted to the IT Department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs must be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 SUPPLEMENTAL INFORMATION FOR TEST

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

25.4.1 Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

25.4.2 Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

25.4.3 Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

25.4.4 Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response must be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP CA-L-S-002, Subcontracting.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of the TestAmerica network are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 CLIENT CONFIDENTIALITY

In situations involving the transmission of environmental test results by telephone, facsimile, or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the client or any other person designated by the client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

"Confidentiality Notice: The information contained in this message is intended only for the use of the addressee, and may be confidential and/or privileged. If the reader of this message is not the intended recipient, or the employee or agent responsible to deliver it to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify the sender immediately."

25.7 FORMAT OF REPORTS

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 AMENDMENTS TO TEST REPORTS

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

When the report is re-issued, a notation of "report reissue" is placed on the cover/signature page of the report or at the top of the narrative page with a brief explanation of reason for the reissue and a reference back to the lst final report generated.

25.9 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS

25.9.1 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

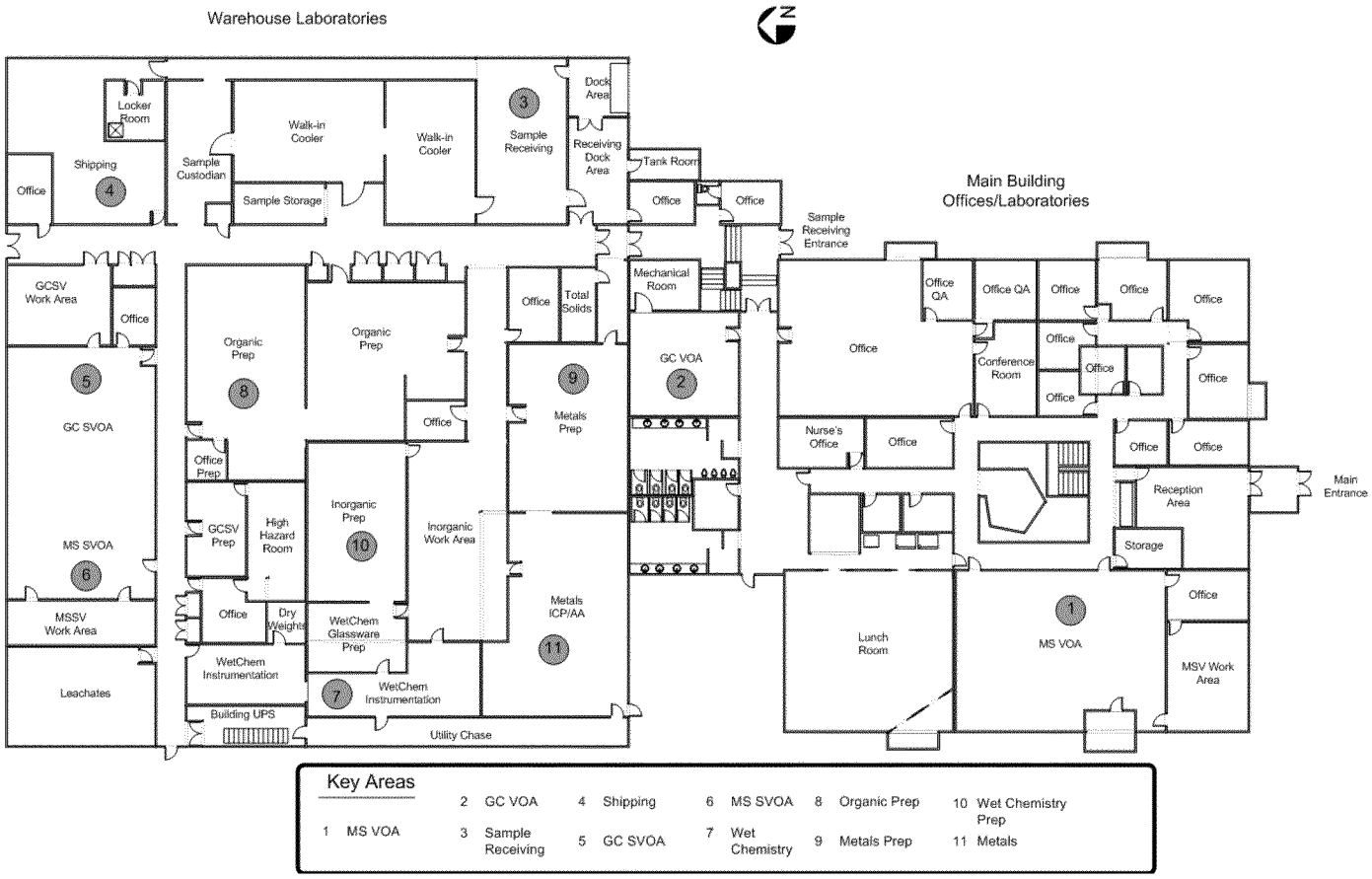
Appendix 1.

Laboratory Floor Plan

TestAmerica – North Canton

4101 Shuffel Dr NW
North Canton, OH 44720

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING



Appendix 2. Laboratory Method Listing

Wet Chemistry Methods[1]

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Acidity	Water	305.[2]	--	SM 2310 B
Alkalinity, Bicarbonate, Carbonate	Water	310.1.[2]		SM 2320 B
	Solid	EPA 310.1[2] (M)	--	--
Arsenic (ASV) Anodic Stripping Voltammetry	Water	--	EPA 7063	--
Fraction of Organic Carbon	Solid	--	--	ASTM D29-74
Biochemical Oxygen Demand, Carbonaceous	Water	EPA 405.1	--	SM 5210 B
Anions, Bromide, Chloride, Fluoride, Sulfate, Nitrite, Nitrate, ortho-phosphate	Water	EPA 300.0	EPA 9056A	--
	Waste	EPA 300.0	EPA 9056A	--
	Solid	EPA 300.0 (M)	EPA 9056A	--
		--		--
Chemical Oxygen Demand	Water	EPA 410.4	--	SM 5220D
	Waste	EPA 410.4	--	--
Chloride	Water	EPA 325.2[2]	EPA 9251	SM 4500 Cl-E
	Solid		EPA 9251(M)	--
Chromium, Hexavalent	Water	EPA 3500-Cr-D	EPA 7196A	SM 3500-Cr-D
	Waste	EPA 3500-Cr-D	EPA 7196A	SM 3500-Cr-D
	Solid	--	EPA 3060A EPA 7196A	--

[1] Any matrix not listed is not applicable for the associated method
[2] Removed from 40CFR

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Specific Conductance	Water	EPA 120.1	EPA 9050A	SM 2510B
	Waste	EPA 120.1	EPA 9050A	--
	Solid	--	EPA 9050A	--
Chlorine, Residual	Water	EPA 330.5[2]	--	SM 4500 CL-G
Cyanide (Amenable)	Water	EPA 335.1[2]	EPA 9012A	SM 4500 CN-G
	Solid	--	EPA 9012A	--
Cyanide (Total)	Water	EPA 335.4	EPA 9012A	SM 4500-CN E 335.2-CLP-M (Ohio VAP)
	Waste	--	EPA 9012A	--
	Solid	--	EPA 9012A	335.2-CLP-M (Ohio VAP)
Cyanide (Weak and Dissociable) (Free)	Water		--	SM 4500-CN I
Dissolved Oxygen	Water	360.1[2]	--	SM 4500 O-G
Flash Point	Waste	--	EPA 1010	
	Solid	--	EPA 1010	
Fluoride	Water	EPA 340.2[2]		SM 4500 F-C, ISE
	Waste	EPA 340.2 (M)[2]		--
	Solid	EPA 340.2 (M)[2]		--
Iron, Ferrous & Ferric	Water		--	SM 3500 FE D
Hardness	Water	EPA 130.2[2]	--	SM 2340B SM 2340C
Moisture	Solid	---	EPA 160.3 (M)	---
Nitrogen, Ammonia	Water	EPA 350.3 EPA 350.2[2]	--	SM 4500 NH_3-E (Titration) SM 4500 NH3-F (ISE)
	Waste	EPA 350.3 EPA 350.2[2]	--	
	Solid	EPA 350.3 EPA 350.2[2]	--	

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Total Kjeldahl Nitrogen (TKN)	Water		EPA 351.3	--	SM 4500 NOrg C
	Waste		EPA 351.3	--	--
	Solid		EPA 351.3	--	--
Oil and Grease (Hexane Extractable Material)	Water		EPA 1664A	EPA 9071B	--
	Waste		EPA 1664A	EPA 9071B	--
	Solid		--	EPA 9071B	--
Ortho-phosphate o-PO_4	Water		EPA 365.1		SM 4500 P-E
	Waste				--
	Solid		EPA 365.1		--
pH	Water		EPA 150.1[2]	EPA 9040B EPA 9040C	SM 4500 H+-B
	Waste			EPA 9045C EPA 9041	SM 4500 H+-B
	Solid		---	EPA 9045C	--
Paint Filter	Water		--	EPA 9095A	--
Phenolics	Water		EPA 420.1	--	--
	Waste		--	EPA 9065	--
	Solid		--	EPA 9065	--
Phosphorus (Total)	Water		EPA 365.1	--	SM 4500 P-E
	Waste		EPA 365.1	--	--
	Solid		EPA 365.1	--	--
Sulfate (SO_4)	Water		EPA 375.4[2]	EPA 9038	--
	Waste		EPA 375.4[2]	EPA 9038	--
	Solid			EPA 9038 (M)	--

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA	Other
Sulfide	Water	EPA 376.1[2]	9030B/9034	SM 4500 S2-E
Total Organic Carbon (TOC)	Water	EPA 415.1[2]	EPA 9060	SM 5310 C
	Waste	--	EPA 9060	--
	Solid	EPA 415.1 (M)	EPA 9060 (M)	Walkley-Black
Total Petroleum Hydrocarbons	Water	EPA 1664A (SGT-HEM)	EPA 9071B	--
	Waste	EPA 1664A (SGT-HEM)	EPA 9071B	--
	Solid	--	EPA 9071B	--
Total Solids	Water	EPA 160.3	--	--
	Waste	EPA 160.3	--	--
	Solid	EPA 160.3 (M)	--	--
Total Dissolved Solids	Water	EPA 160.1	--	SM2540C
Total Suspended Solids	Water	EPA 160.2	---	SM2540D
Volatile and Volatile Suspended Solids	Water	EPA 160.4	--	--
Settleable Solids	Water	EPA 160.5	--	SM2540F
Turbidity	Water	EPA 180.1	--	--
Specific Gravity	Water			SM 2710F

Methods for Mercury by Cold Vapor Atomic Absorption

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Mercury (CVAA)	Water	EPA 245.1	EPA 7470A	--
	TCLP Leachate	--	EPA 7470A	--
	Waste	--	EPA 7471A, 7471B	--
	Solid		EPA 7471A, 7471B	--

Methods for Mercury by Cold Vapor Atomic Fluororescence

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Mercury, Low Level (CVAFS)	Water	--	--	EPA 1631E
	Solid	--	--	EPA 1631E

Methods for Metals by ICP and ICPMS

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Metals by ICP analysis	Water	EPA 200.7	EPA 6010B, 6010C	---
	Waste	---	EPA 6010B, 6010C	---
	Solid	EPA 200.7	EPA 6010B, 6010C	---

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Metals by ICPMS analysis	Water		EPA 200.8	EPA 6020, 6020A	---
	Waste		---	EPA 6020, 6020A	---
	Solid		EPA 200.8	EPA 6020, 6020A	---

Metals Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Toxicity Characteristic Leaching Procedure (TCLP)/ SPLP Extraction	Water	---	EPA 1311 EPA 1312	---
	Waste	---	EPA 1311 EPA 1312	---
	Solid	---	EPA 1311 EPA 1312	---
ICP Metals	Water	EPA 200.7	EPA 3005A EPA 3010A	---
	TCLP Leachate	---	EPA 3010A	---
	Waste	---	EPA 3050B	---
	Solid	---	EPA 3050B	---
ICPMS Metals	Water	EPA 200.8	EPA 3010A	---
	TCLP	---	EPA 3010A	---
	Waste	---	EPA 3050B	---
	Solid	---	EPA 3050B	---
CVAA Mercury	Water	EPA 245.1	EPA 7470A	---
	TCLP Leachate	---	EPA 7470A	---
	Waste	---	EPA 7471A EPA 7471B	---
	Solid	---	EPA 7471A EPA 7471B	
CVAFS Mercury Low Level	Water	---	---	EPA 1631E
	Solid	---	---	EPA 1631E

Organic Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water	EPA 624	EPA 5030B EPA 5030C	---
	Waste	---	EPA 5030B EPA 5030C EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Semivolatiles by GC/MS	Water	EPA 625	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3550C EPA 3540C EPA 3580A	---
	Solid	---	EPA 3550B EPA 3550C EPA 3540C	---
Pesticides/PCBs by GC	Water	EPA 608	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	EPA 3510C EPA 3520C	---

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
	Waste		---	EPA 3550B EPA 3550C EPA 3540C EPA 3546 (PCB only) EPA 3580A	---
	Solid		---	EPA 3550B EPA 3550C EPA 3540C	---

Analytical Parameters	Matrix		Fields of Testing		
			CWA RCRA (SW846)		Other
Herbicides by GC	Water			EPA 8151A	---
	Waste		---	EPA 8151A	---
	Solid		---	EPA 8151A	---
Total Petroleum Hydrocarbons (Gasoline Range) by GC	Water		---	EPA 5030B EPA 5030C	WI GRO
	Waste		---	EPA 5030B EPA 5030C EPA 5035 EPA 5035A	WI GRO
	Solid		---	EPA 5035 EPA 5035A	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC	Water		---	EPA 3510C EPA 3520C	WI DRO
	TCLP Leachate		---	EPA 3510C EPA 3520C	---
	Waste		---	EPA 3550B EPA 3550C EPA 3580A	WI DRO
	Solid		---	EPA 3550B EPA 3550C	WI DRO

Organic Methods of Analysis

Analytical Parameters	Matrix		Fields of Testing		
			CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water		EPA 624	EPA 8260B EPA 8260C	---
	Waste		---	EPA 8260B EPA 8260C	---
	Solid		---	EPA 8260B EPA 8260C	---
Semivolatiles by GC/MS	Water		EPA 625	EPA 8270C EPA 8270D	
	Waste		---	EPA 8270C EPA 8270D	---
	Solid		---	EPA 8270C EPA 8270D	---
Pesticides/PCBs by GC	Water		EPA 608	Pesticides 8081A, 8081B PCBs 8082, 8082A	---
	TCLP Leachate		---	Pesticides 8081A, 8081B PCBs 8082, 8082A	---
	Waste		---	Pesticides 8081A, 8081B PCBs 8082, 8082A	---
	Solid		---	Pesticides 8081A, 8081B PCBs 8082, 8082A	---

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Phenoxyacid Herbicides by GC	Water	---	EPA 8151A	---
	TCLP Leachate	---	EPA 8151A	---
	Waste	---	EPA 8151A	---
	Solid	---	EPA 8151A	---
Gasoline Range Organics by GC	Water	---	EPA 8015B (M) EPA 8015C	WI GRO
	Waste	---	EPA 8015B (M) EPA 8015C	---
	Solid	---	EPA 8015B (M) EPA 8015C	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC/FID	Water	---	EPA 8015B (M) EPA 8015C	WI DRO
	Waste	---	EPA 8015B (M) EPA 8015C	---
Dissolved Gases RSK-175	Water	---	---	SOP
Formaldehyde Carbonyl Compounds	Water	---	EPA 8315A	---
	Solid	---	EPA 8315A	---
Aromatic Acids	Water	---	---	SOP
	Solid	---	---	SOP
Methyl Mercury	Water	EPA 1630	---	---
	Solid	EPA 1630	---	---

Appendix 3. Glossary/Acronyms

Glossary

Acceptance Criteria:
Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQ)

Accreditation:
The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy:
The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst:
The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Uncertainty:
A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Assessment:
The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit:
A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch:

Environmental samples which are prepared and/or anal yzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples. (TNI)

Bias:
The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank:
A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQ)

Calibration:
A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve:
The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard:
A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM):
A reference material accompanied by a certificate having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute.

Chain-of-Custody:
Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes the number and types of containers, the mode of collection, the collector, time of collection, preservation, and requested analyses. (TNI)

Compromised Samples:
Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI):
Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. TNI and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation:
Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:Second column confirmation
Alternate wavelength
Derivatization
Mass spectral interpretation
Alternative detectors or
Additional cleanup procedures

Conformance:
An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction:
Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis . No significant action is taken to change behavior, process or procedure.

Corrective Action:
The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit:
A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction:

The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors and collation into a more useable form. (TNI)

Deficiency:
An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Demonstration of Capability:
A procedure to establish the ability of the anlayst to generate anlytical results of acceptable accuracy and precision. (TNI)

Document Control:
The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQ)

Duplicate Analyses:
The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank:
Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration:
Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Field Blank:
Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation:
Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times:
The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard:
A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration:
Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank:
A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL):
The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is + _ 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):
A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS must be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total

solids, pH, color, odor, temperature, dissolved oxyg en or turbidity. The results of these samples must be used to determine batch acceptance.

Least Squares Regression (1st Order Curve):
The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit(s) of Detection (LOD) (a.k.a., Method Detection Limit [MDL]):
A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliabl detect in their facility. (TNI)

LOD Verification (a.k.a., MDL Verification):
A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]:
The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (TNI)

(QS) Matrix:
The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions must be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with ,15% settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples must be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with .15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air and Emissions: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

Matrix Spike (spiked sample or fortified sample):
A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate):
A replicate matrix spike is prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.
Method Blank:
A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit:
The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

Negative Control:
Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance:
An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Performance Audit:
The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control:
Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision:
The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation:
Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Proficiency Testing:
A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program:
The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT):
A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance:
An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP):
A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control:
The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample:

A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual:
A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System:
A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Raw Data:
The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention:
The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material:
A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Method:
A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

Reference Standard:
A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2nd order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity:
The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Spike:
A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard:
The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs):
A written document which details the method of an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPS are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate:
A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and must be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit):

A thorough, systematic, qualitative on-site assessme nt of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty:
A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms

ASTM	American Society for Testing & Materials
CAR	Corrective Action Report
CBI	Confidential Business Information
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CF	Calibration Factor
CFR	Code of Federal Regulations
COC	Chain of Custody
CQMP	Corporate Quality Management Plan
CSM	Customer Service Manager
DOC	Demonstration of Capability
DoD	Department of Defense
DQO	Data Quality Objectives
DUP	Duplicate
ECO	Ethics and Compliance Officer
EDD	Electronic Data Deliverable
EHS	Environment, Health and Safety
EPA	Environmental Protection Agency
GC	Gas Chromatography
GC/MS	GasChromatography/Mass Spectrometry
HPLC	High Performance Liquid Chromatography
ICP	Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS	ICP/Mass Spectrometry
ICB	Initial Calibration Blank
ICV	Initial Calibration Verification
IDL	Instrument Detection Limit
IEC	International Electrotechnical Commission
IS	Internal Standard
ISO	International Organization for Standardization
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOD	Limit of Detection
LOQ	Limit of Quantitation
LIMS	Laboratory Information Management System
MDL	Method Detection Limit
MDLCK	MDL Check Standard
MDLV	MDL Verification Check Standard
MRL	Method Reporting Limit Check Standard
MS	Matrix Spike
MSD	Matrix Spike Duplicate

MSDS Material Safety Data Sheet	
NCM Nonconformance Memo	
NELAP National Environmental Laboratory Accreditation Program	
NIST National Institute of Standards and Technology	
NPDES National Pollutant Discharge Elimination System	
OVAP Ohio Voluntary Action Program	
PM	Project Manager
PT	Performance Testing
TIC	Tentatively Identified Compound
TNI	The NELAC Institute
QAM Quality Assurance Manual	
QA/QC Quality Assurance / Quality Control	
QAPP Quality Assurance Project Plan	
RCRA Resource Conservation and Recovery Act	
RF	Response Factor
RFP	Request for Proposal
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SAP	Sampling and Analysis Plan
SD	Standard Deviation
SOP	Standard Operating Procedure
SPLP SPLP = Synthetic Precipitation Leaching Procedure	
TAT	Turn-Around Time
TCLP Toxicity Characteristic Leaching Procedure	
TSCA Toxic Substances Control Act	
USACE United States Army Corps of Engineers	
USDA United States Department of Agriculture	
VOA	Volatiles

Appendix 4. Laboratory Certifications, Accreditations, Validations

TestAmerica North Canton maintains certifications, accreditations, certifications, and approvals with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Organization	Certificate Number	Organization	Certificate Number
California	01144CA	Nevada	OH-00048208A
Connecticut	PH-0590	New Jersey	OH001
Florida	E87225	New York	10975
Georgia	---	OVAP	CL0024
Illinois	001298	Pennsylvania	68-00340
Kansas	E-10336		
Kentucky Underground Storage Tank Program	0058	USDA (Dept.of Agriculture)	P330-08-00123
Minnesota	039-999-348	West Virginia	210
DoD – LAB	L2315	Wisconsin	999518190

The certificates and parameter lists (which may differ) are available, upon request, from a laboratory representative for each organization may be found on the corporate web site, the laboratory's public server, the final report review table, and in the following offices: QA, Marketing, and Project Management.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Canton

Policy No. QA-003, Rev. 12
Effective Date: 10/31/13

Title: QUALITY CONTROL PROGRAM

Approvals (Signature/Date):

Rebecca Strait 10/28/13
Quality Assurance Manager Date

10/30/13
Laboratory Director Date

This SOP was previously identified as Policy No. QA-003, Rev 11, dated 04/30/12

Facility Distribution No. _____ **Distributed To: _____**

Table of Contents

Attachments

1. OBJECTIVE

1.1. This policy describes the TestAmerica Canton program of routine analytical quality control (QC) activities. The objective is to generate QC data that demonstrates that the analytical process is in control and that the data meets client and method requirements. The policy outlines QC requirements for a variety of regulatory programs, with the stipulation that when lacking specific direction from our clients, TestAmerica Canton will default to routine RCRA program QC requirements.

2. SCOPE

2.1. This policy is to be enforced and followed throughout the laboratory.

3. POLICY

3.1. Assessments of QC data relative to control limits determine the acceptability of sample test results. Whenever control criteria are not met, the data must be evaluated to determine appropriate corrective action. The initial evaluation is made by the analyst, frequently in conjunction with data review software and/or senior analysts or supervisors. Further technical evaluation of the data or data review software output is conducted by second-party data reviewers. Corrective action decisions, particularly whether or not to reanalyze samples, should be done in consultation with the client to the extent possible when operating under project-specific QA plans. Requirements for assessment and corrective action are described in the attachments to this policy. Details concerning technical data review and documentation of the reviews are described in the TestAmerica Canton Quality Assurance Manual (QAM), current version.

3.2. The TestAmerica Canton standard QC program is to be communicated to the client prior to acceptance of work. At the same time, every effort must be made to understand the client's special project requirements. Generally, laboratory Project Managers serve as a liaison between the clients and the laboratory staff to ensure that requirements are properly communicated in writing to both parties. In the event that alternative QC procedures are not specified by our clients, these standard QC protocols must be followed to ensure the generation of legally and scientifically defensible analytical data.

3.3. Successful implementation of this QC program requires that it is clearly understood by all TestAmerica staff. Training based on this policy must be conducted

periodically and provided to new personnel as appropriate for their functions.

3.4. TestAmerica Canton QC program applies to the following:

3.4.1. RCRA and SW-846 Projects

3.4.1.1. All routine analytical projects performed using SW-846 methods must comply with the requirements described in the TestAmerica Canton Quality Assurance Manual (QAM) and Attachment I to this policy. The Quality Control sections of analytical standard operating procedures (SOPs) referencing SW-846 methods must be consistent with the requirements in Attachment I.

3.4.2. CWA and 40 CFR Part 136 Projects

3.4.2.1. Any analytical work conducted in support of an NPDES permit or other Clean Water Act compliance activities, must meet the quality control specifications shown in the TestAmerica Canton Quality Assurance Manual (QAM). The quality control requirements for the specific methods listed in the QAM define the minimum requirements that must be given in laboratory analytical SOPs.

3.4.3. Other Programs or Projects with Clearly Defined QC Requirements

3.4.3.1. The differences between the TestAmerica Canton standard QC program and special project requirements must be specified in project documents. These documents may include Quality Assurance Project Plans (QAPPs), Sampling and Analysis Plans (SAPs), project-specific Quality Assurance Summaries (QASs), SOPs, contracts, or other approved documents.

3.4.3.2. Documents describing special project requirements must be reviewed and approved by the Laboratory Director, QA, Project Management, and Operations as appropriate.

3.4.3.3. If the special project requirements appear to result in modifications that contradict federal or state regulatory requirements, the variance must be noted in writing and communicated to the client. A record of this communication must be retained as a permanent part of the project file.

3.4.3.4. Any special client project requirements must be communicated to TestAmerica Canton's analysts in advance of releasing samples for analysis, and the work must be clearly differentiated in the analytical documentation, otherwise Attachment I requirements must be followed.

3.4.4. Projects Without Specific QC Requirements

3.4.4.1. Any projects for which no specific QC program is specified must follow the requirements shown in Attachment I.

3.5. Analytical SOPs must include a quality control section that addresses these general QC requirements. As relevant, specific method QC requirements should be given precedence to these general requirements.

4. REVISION HISTORY

Historical File:		Revision 2: 07/13/98		Revision 8: 03/18/10
		Revision 3: 10/11/01		Revision 9: 03/11/11
		Revision 4: 10/03/02		Revision 10: 04/13/11
		Revision 5: 09/27/04		Revision 11: 04/30/12
		Revision 6: 09/19/07		
		Revision 7: 09/30/08		

5. REFERENCES

5.1. TestAmerica Canton Quality Assurance Manual (QAM), current version

5.2. Corporate Quality Management Plan (CQMP), current version

5.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

5.4. Supplemental Practices for DoD Project Work, NC-QA-016

5.5. Statistical Evaluation of Data & Development of Control Charts, NC-QA-018

5.6. Nonconformance and Corrective Action, NC-QA-029

ATTACHMENT I

QC FOR RCRA PROJECTS AND PROJECTS WITHOUT DEFINED QC REQUIREMENTS

1. INTRODUCTION

1.1. This Quality Control (QC) Program is based on the requirements in "Test Methods for Evaluating Solid Waste", USEPA SW-846. It applies whenever SW-846 analytical methods are used. It also applies in whole or in part whenever project requirements fail to specify some aspect of QC practices described here. It does not apply when other well-defined QC programs (e.g., DoD QSM or Ohio VAP) are specified. This policy represents TestAmerica Canton base QC program for environmental analyses.

1.2. Details concerning instrument calibrations, tunes, and QC that are required for specific methods (e.g., interference check samples for ICP) are not given here. Refer to the method standard operating procedures (SOPs) for information about the frequency, assessment and corrective action required for additional QC elements.

2. DEFINITIONS

2.1. Batch Definition:

2.1.1. A batch is a group of no greater than 20 samples, excluding QC samples (LCS, Method Blank, MS, and MSD) which are processed similarly with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

2.2. Surrogates:

2.2.1. Surrogates are organic compounds similar in chemical behavior to the target analytes, but that are not normally found in environmental samples. Surrogates are added to all samples in a batch to monitor the effects of both the matrix and the analytical process on accuracy.

2.3. Method Blank (MB):

2.3.1. The MB is a control sample prepared using the same reagents used for the samples. As part of a QC batch, it accompanies the samples through all steps of the analytical procedure. The MB is used to monitor the level of contamination introduced to a batch of samples as a result of laboratory processing.

2.4. Instrument/CalibrationBlank:

2.4.1. The instrument blank is prepared using the same solvents and reagents (e.g. hexane, methylene chloride, or reagent water) used to dilute the prepared sample extracts or digests. Unlike the MB, it is analyzed without being subject to the preparation steps of the analytical procedure. It is used to monitor laboratory or reagent contamination introduced at the instrumental analysis phase of work. For procedures without a separate preparation step, an instrument blank is equivalent to the MB, and serves the same purpose.

2.5. Laboratory Control Sample (LCS):

2.5.1. The LCS is prepared using a well characterized matrix (e.g., reagent water or Ottawa sand) that is spiked with known amounts of representative analytes. Alternate matrices (e.g., glass beads) may be used for soil analyses when Ottawa sand is not appropriate. As part of a QC batch, it accompanies the samples through all steps of the analytical process. The LCS is used to monitor the accuracy of the analytical process independent of possible interference effects due to sample matrix. Information regarding precision of the method can be determined over time.

2.6. Matrix Spike and Matrix Spike Duplicate (MS/MSD):

2.6.1. Matrix Spike - A matrix spike (MS) is a replicate portion of one field sample in the QC batch that is spiked with known amounts of target analytes. An MS is spiked with the same analytes at the same concentrations that are added to the LCS. Any client sample that is not a field blank or an equipment blank can be used for a matrix spike as long as there is sufficient quantity. As part of the QC batch, it accompanies the field samples through all steps of the analytical process. Matrix spike data are only meaningful for the sample in which they are prepared and samples from the same site.

2.6.2. Matrix Spike Duplicate: A matrix spike duplicate (MSD) consists of an additional portion of the same sample used to prepare the MS. This portion is spiked and processed exactly as the MS.

2.6.3. The MS and MSD results are used to determine the effect of the sample matrix on the precision and accuracy of results. Due to the potential variability of the matrix of each sample, the MS and MSD results may not have immediate bearing on any samples except the one spiked.

2.7. Sample Duplicate (DU):

2.7.1. A DU is a second aliquot of an environmental sample, taken from the same sample container when possible, that is processed with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. Any client sample that is not a field blank or equipment blank can be used for a DU as long as there is sufficient quantity. The sample and duplicate results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample duplicate precision results are not necessarily representative of the precision for other samples in the batch.

2.8. Duplicate Control Sample (LCSD):

2.8.1. A duplicate laboratory control sample (LCSD) may be required by specific client or regulatory programs.

3. BATCH QC ELEMENTS & BATCH PROCESSING

3.1. A QC batch is designed to determine the quality of the analytical results obtained for a group of up to 20 field samples in terms of accuracy and precision. With some exceptions as described in Sections 3.6 through 3.8 below, the minimum QC elements for each QC batch are:

3.1.1. One method blank (MB)

3.1.2. One laboratory control sample (LCS)

3.1.3. One matrix spike (MS)

3.1.4. One matrix spike duplicate (MSD)

3.2. The identity of each QC batch must be documented and traceable, i.e., each batch of field samples must be clearly associated with the applicable QC samples.

3.3. To the extent possible, samples that require a preparation step should be analyzed together with their associated QC samples

3.4. For analytical procedures that do not include a separate extraction or digestion (e.g., volatile organic analysis by purge and trap), the QC batch must be analyzed sequentially using the same instrument and instrument configuration within the same calibration event. That is, the same calibration curve, calibration factors, or response factors must be in effect throughout the analysis.

3.5. Field QC samples (e.g., trip blanks, equipment rinsates, and field duplicates) count as individual samples, therefore, they add to the QC batch count. Samples that require simple re-analysis (e.g., dilutions to adjust a sample extract to the working range of the instrument), as opposed to re-extraction or digestion and re-analysis, do not count as additional samples in the QC batch. For procedures without a separate preparation, a re-analysis within the same calibration event (as defined in Section 3.4) does not add to the batch count.

3.6. MS/MSD pairs are not the only acceptable means of demonstrating precision.

3.6.1. As requested by clients or required by some methods, batch precision may also be demonstrated through the analysis of sample duplicates. However, the client should be advised that a duplicate is less likely to provide usable precision statistics depending on the likelihood of finding concentrations below reporting limits.

3.6.2. Ongoing monitoring of LCS results can be used to determine long-term precision and accuracy for a method.

3.7. Some methods including isotope-dilution methods, pH and ignitability, for example, do not use all of the QC elements listed in Section 3.1. Method exceptions to these requirements are listed in the TestAmerica Canton Quality Assurance Manual (QAM) QC tables and in the laboratory analytical SOPs.

3.8. Deviations from these QC elements must either be noted in project planning documents (QAPPs, SAPs, SOWs, QAS, or equivalent) or in a nonconformance memo (see SOP NC-QA-029 for details).

4. DATA EVALUATION AND CORRECTIVE ACTION

4.1. General Guidelines

4.1.1. Any QC component that is outside of established control limits is considered an out-of-control event. All out-of-control events must be documented and the associated data evaluated. Depending on the specific circumstances, evaluation can lead to a variety of actions. The following sections and the flowcharts describe the appropriate corrective action for the most common QC failures. However, it is not possible to address all possible data evaluation scenarios in this policy. The guiding principle for all evaluations is that the data and corrective action decisions must be defensible using TestAmerica Canton policies, procedures or scientific evidence, and justified in the project records.

4.1.2. If re-analysis for QC failures is conducted and the second analysis confirms a QC problem that is outside of the laboratory's control, further testing is not necessary. The problem must be documented and the data properly qualified in the project report.

4.1.3. QC failures that are not corrected by re-analysis are documented in the TestAmerica Canton electronic Nonconformance System as described in SOP NC-QA-029.

4.1.4. QC failures due to sample matrix interferences (particularly MS, MSD, and sample surrogate failures) must be documented in either the NCM system or by use of a Data Review Checklist. All holding time violations MUST be documented in the NCM system. In all cases, matrix QC failures must be communicated to the laboratory Project Manager and significant matrix QC failures must be discussed in the final report case narrative.

4.1.5. When ongoing systematic problems are identified, work may stop until it can be demonstrated that the system is in control again.

4.2. Method Blank (MB) Evaluation (see Figure 1)

4.2.1. Method Blank Acceptance Criteria

4.2.1.1. The result of the MB is one of the QC measures used to assess batch acceptance. Results are acceptable if all analyte concentrations in the MB meet the following criteria:

4.2.1.1.1. Organics: The blank contamination is less than 1/10 of the measured concentration of any sample in the associated preparation batch, or

4.2.1.1.2. Inorganics: The blank contamination is less than 1/10 of the measured concentration of any sample in the associated preparation batch, or

4.2.1.1.3. The blank contamination is less than the concentration present in the samples and is less than 1/10 of the regulatory limit, or

4.2.1.1.4. The same contaminants were not found in the associated samples, or

4.2.1.1.5. MB results are less than or equal to the reporting limit

Note: Positive MB results slightly below the reporting limit should still be evaluated by the analyst for potential impact on sample results at or near the reporting limit.

Note: DoD project requirements are noted in NC-QA-016, Supplemental Practices for DoD Project Work.

Note: For Ohio VAP projects, the MB contamination must be below the reporting limit, unless the associated sample is less than the reporting limit.

4.2.2. Corrective Action for MB Failure

4.2.2.1. If the MB does not meet the acceptance criteria, the source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem. Samples associated with the contaminated blank must be re-processed for analysis, or under the following circumstances, may be reported as qualified (qualifier flags or narrative comments):

4.2.2.1.1. MB contamination at a level less than the reporting limit with sample results at levels near the RL, based on analyst's judgement shall be flagged, if flags are requested by client.

4.2.2.1.2. Analyte concentrations in samples are greater than 10 times blank contamination, or

4.2.2.1.3. The contaminant is a common blank contaminant (see below) and the MB concentration is less than five times the RL for organics or less than two times the RL for inorganics. Note that some programs do not recognize common lab contaminants.

Common Laboratory Contaminants

Analyte	Method
Methylene Chloride	Volatile Organics (GC or GC/MS)
Acetone	Volatile Organics (GC or GC/MS)
2-Butanone	Volatile Organics (GC or GC/MS)
Chloroform	Volatile Organics (GC or GC/MS, SPLP only)
Phthalate Esters	Semi-Volatile Organics (GC or GC/MS)
Copper	Metals (ICP or ICPMS)
Zinc	Metals (ICP or ICPMS)
Iron	Metals (ICP or ICPMS)
Lead	Metals (ICP or ICPMS)
Barium	Metals (ICPMS)
Chromium	Metals (ICPMS)

Analyte	Method
Manganese	Metals (ICPMS)
Calcium	Metals (ICPMS)
Magnesium	Metals (ICPMS)
Potassium	Metals (ICPMS)
Sodium	Metals (ICPMS)

4.3. Laboratory Control Samples (LCS) Evaluation (see Figure 2)

4.3.1. Acceptance Criteria

4.3.1.1. The LCS recovery for the control analytes must be within established control limits. The percent recovery is calculated as follows:

$$\text{LCS Percent Recovery} = \frac{X}{t} \times 100$$

Where: X = observed concentration
t = concentration of spike added

4.3.2. Corrective Action for LCS Failure

4.3.2.1. Check calculations

4.3.2.2. Check instrument performance

4.3.2.3. Re-analyze the LCS, and if still outside of control limits

4.3.2.4. Re-prepare and re-analyze all samples in the QC batch

Notes:

1. It is acceptable to report the data if the LCS recovery is out high and analyte of concern was not detected in any of the samples.

2. In the case of volatile analyses, if the LCS fails, a new LCS may be re-prepared and re-analyzed within the same tune period.

3. In the case where all target requested analytes are within control, but some other LCS compounds are out of control, the LCS may still be considered acceptable for reporting.

4.4. Duplicate Laboratory Control Samples (LCSD) Evaluation (see Fig. 2)

4.4.1. Acceptance Criteria

4.4.1.1. The recovery for each spike of the pair must be within established control limits as noted in specific state or client requirements. The formula used to calculate LCSD recoveries is the same as the formula for LCS spike recoveries.

4.4.1.2. The relative percent difference (RPD) for the pair is calculated as follows:

$$\text{RPD} = \left[\frac{|X_1 - X_2|}{\frac{(X_1 + X_2)}{2}}\right] \times 100$$

Where: X_1 = first observed concentration

X_2 = second observed concentration

4.4.2. Corrective Action for LCS/LCSD Recovery (Accuracy)Failure

4.4.2.1. Corrective action for LCSD failure is the same as the LCS as noted in Section 4.3.2. Both the LCS and LCSD must meet acceptance criteria.

4.5. Surrogate Evaluation (see Figure 3)

4.5.1. Acceptance Criteria

4.5.1.1. Surrogate recoveries must be within established control limits. Method QC (MB or LCS) results are not acceptable unless the surrogate recoveries for those QC samples are within control limits. If MS/MSD, duplicate, or field samples require dilutions beyond the threshold stated in the analytical SOPs, routine surrogate control limits do not apply and recoveries are not evaluated. This should be noted in the final report. The recovery is calculated as follows:

$$\text{Surrogate Percent Recovery} = \frac{X}{t} \times 100$$

Where: X = observed concentration

t = concentration of surrogate added

4.5.2. Corrective Action

4.5.2.1. Surrogate Failures in MB or LCS

4.5.2.1.1. Check calculation and instrument performance

4.5.2.1.2. Re-analyze QC sample and/or re-analyze all samples in the QC batch

Note: For Ohio VAP projects, the batch must be re-extracted if re-analysis does not resolve the problem.

4.5.2.2. Surrogate Failures in Samples or MS/MSD

4.5.2.2.1. Check calculation and instrument performance

4.5.2.2.2. Evaluate objective evidence of matrix interference (e.g., heterogeneous sample, interfering compounds seen on chromatograms, or interference demonstrated by prior analyses)

4.5.2.2.3. Document the failure and note it on the final report

4.5.2.2.4. If samples require dilutions beyond the threshold stated in the analytical SOPs, routine control limits do not apply and recoveries are not evaluated. This should be noted in the final report.

Note: Unless otherwise specified by the client, it may be possible to report qualified results if method QC surrogate recoveries are biased high and analytes were not detected in the field samples. However, all other QC requirements would have to be met and the failure would have to be noted in the final report.

Note: Some client programs require re-analysis to confirm matrix interferences. Check special project instructions for this corrective action.

4.6. Matrix Spike and Matrix Spike Duplicates (MS/MSD) Evaluation (see Figure 4)

4.6.1. Acceptance Criteria

4.6.1.1. MS and MSD recoveries and RPD should be within established control limits.

4.6.1.1.1. If MS or MSD samples require dilutions beyond the threshold stated in the analytical SOPs, routine control limits do not apply and recoveries are not evaluated, but this should be noted in the final report. The MS and MSD recoveries are calculated as follows:

$$\text{MS or MSD Percent Recovery} = \left[\frac{X_s - X}{t}\right] \times 100$$

Where: X = observed concentration in unspiked sample
X_s = observed concentration in spiked sample
t = concentration of spike added

Note:

1. If sample result is ND, X = 0 when no values reported below RL.

2. If sample result is reported as a value <RL, X = reported value.

4.6.1.2. RPD is defined in Section 4.4.1.

4.6.2. Corrective Action for MS/MSD or MS/MSD RPD Failure (assuming that the LCS is in control)

4.6.2.1. Check calculation and instrument performance

4.6.2.2. Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering compounds seen on chromatograms, or interference demonstrated by prior analyses).

4.6.2.3. Document the failure and note on final report;

Note: Some client programs require re-analysis to confirm matrix interferences. Check special project requirements for this corrective action.

4.7. Sample Duplicate

4.7.1. Acceptance Criteria

4.7.1.1. The RPD for the sample and its duplicate must be within established control limits. The RPD is the same as for the MS/MSD (see Section 4.6.1).

4.7.2. Corrective Action for Duplicate Failure

4.7.2.1. Check calculation and instrument performance

4.7.2.2. Document the QC failure and note on the final report

5. ESTABLISHING QC ACCEPTANCE LIMITS

5.1. Initial Control Limits

5.1.1. For new procedures, published method limits can be used until sufficient QC data are acquired. A minimum of 20 to 30 data points are recommended. However, the published limits may not be appropriate if they are based on a single-operator or single-laboratory study. In this case, the QA Manager may establish default limits until enough data is collected for laboratory established limits to be determined.

5.2. Control limits should be re-examined periodically, and reset as needed. If the recalculated limits are consistent with the historical limits, the historical limits may remain unchanged.

5.3. Running the Control Limits Program

5.3.1. Evaluating control charts is an important first step in considering new control limits. This is done with a Control Limit program in LIMS. Only QA personnel are authorized to set control limits. The program collects a specified set of QC data, performs a Grubbs Outlier Test, calculates three standard deviation control limits, compares those limits to the existing limits in the laboratory LIMS, and generates an I-type control chart (ref. ASTM D 6299). This control chart is a plot of results in chronological order to which existing control limits and a centerline have been added. The control chart aids in the examination of the data to be sure that it is representative and appropriate for use in setting new limits. Refer to SOP NC-QA-018, Statistical Evaluation of Data & Development of Control Charts, for complete details; but some specific requirements include the following.

5.3.2. Grubbs Outlier Test

5.3.2.1. The test calculates a value for T, based on the difference of the suspect point from the mean value, quantity divided by the calculated standard deviation.

$$T = \frac{|X_i - \overline{X}|}{s}$$

where: X_i = the point being considered for rejection
$\overline{X}$ = bar is the mean, and
s = the standard deviation

5.3.2.2. The point is rejected if:

$$T > \frac{(N-1)}{\sqrt{N}} \sqrt{\frac{t^2_{(\alpha/2N),N-2)}}{N-2+t^2_{(\alpha/2N),N-2)}}},$$

where: N = number of points
t = t distribution

5.3.2.3. Tables for critical values of T are given in John Taylor, Quality Assurance of Chemical Measurements, Lewis Publishers; 1987. If the measured value of T is greater than the value in the critical value, X_i is rejected. This assumes a normal distribution (see www.itl.nist.gov/div898/handbook/eda for details about the derivation of the critical values of T).

5.4. Examine and Investigate Collected Data

5.4.1. Assuming that an adequate amount of data are collected, the next step involves determining that the data set is representative of the lab's performance, and therefore provides a useful prediction of future performance. A key part of the process is examining the data for bias, discontinuities, and/or trends. Ideally, if conditions are constant over the time period selected and existing limits are appropriate, the data will be evenly distributed around the centerline, with a few points at or slightly outside control limits. The reasons for deviations from the ideal should be investigated to be sure that the collected data are appropriate. Specific conditions requiring further investigation include data sets with no outliers, data with significant bias relative to existing limits, excessive number of outliers, discontinuous patterns, and upward or downward sloping trends.

5.5. 5.5 Selecting New Control Limits

5.5.1. Generally, control limits are based on the following statistics for the historical data:

Accuracy: mean recovery $\pm 3s$
Precision: zero to (mean RPD + 3s)

Where: s = standard deviation

5.5.1.1. If the calculated three standard deviation limits are tighter than the method calibration verification criterion (e.g., CCV acceptance limits for ICP = ± 10% of expected value), then the new limits are set to the mean value ± calibration criterion.

5.6. Communicating and Implementing New Control Limits

5.6.1. QA personnel prepare control limit reports comparing the new control limits with the old. This information is sent to the supervisor of the area affected by the new limits. The supervisor is to review the summary data and confirm that the data selected are representative of current performance. The supervisor is also confirming that the instrument data systems will be updated on the implementation date--the same date that LIMS will be updated.

6. REPORTING QC DATA

6.1. QC data routinely reported with sample results include the LCS, method blank and surrogate standards. Client reporting format requirements are negotiable and documented as part of the project records. Ultimately, all reporting decisions should accommodate the client's requirements.

Figure 1 - Method Blank Evaluation

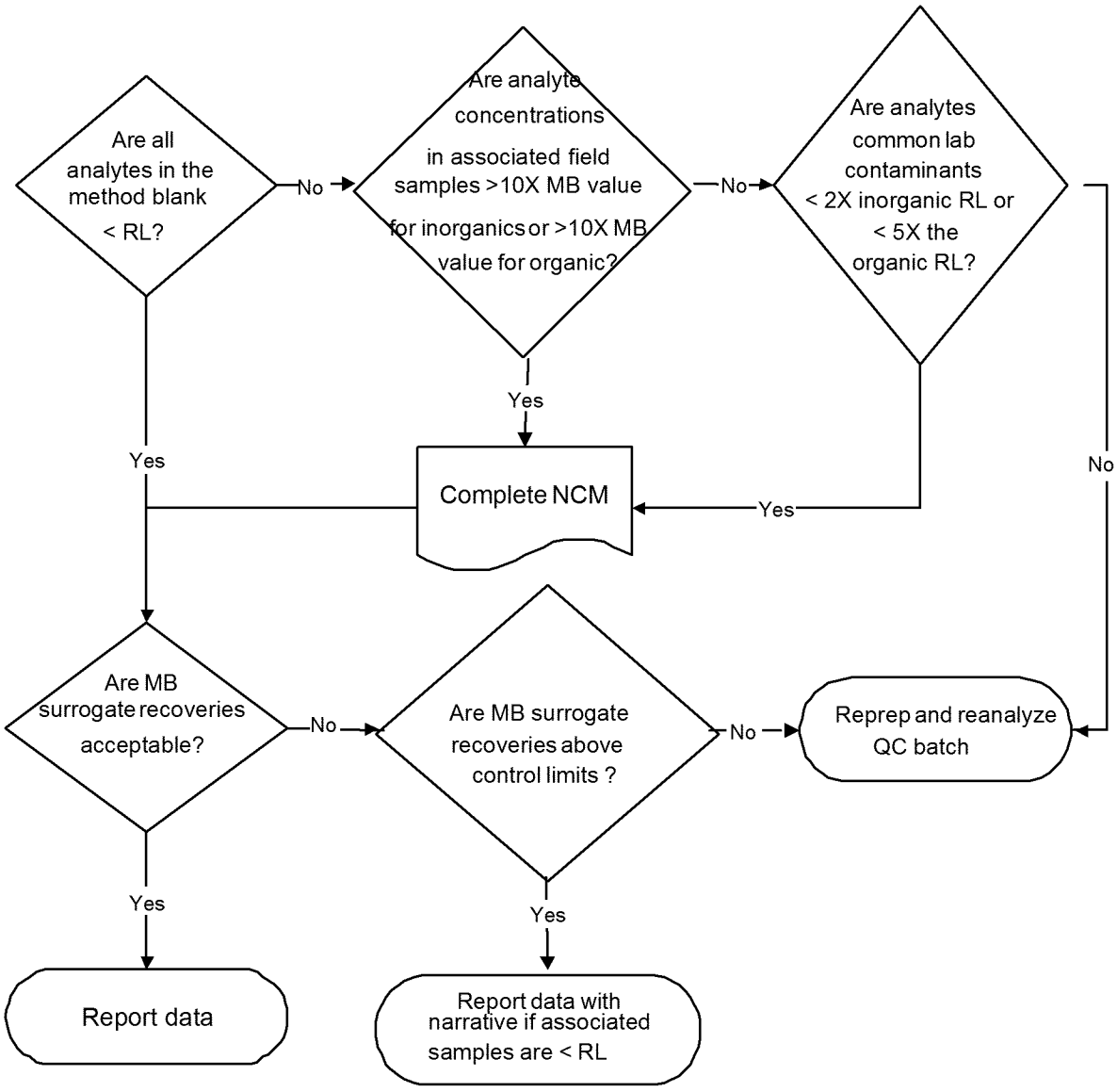


Figure 2 – LCS Evaluation

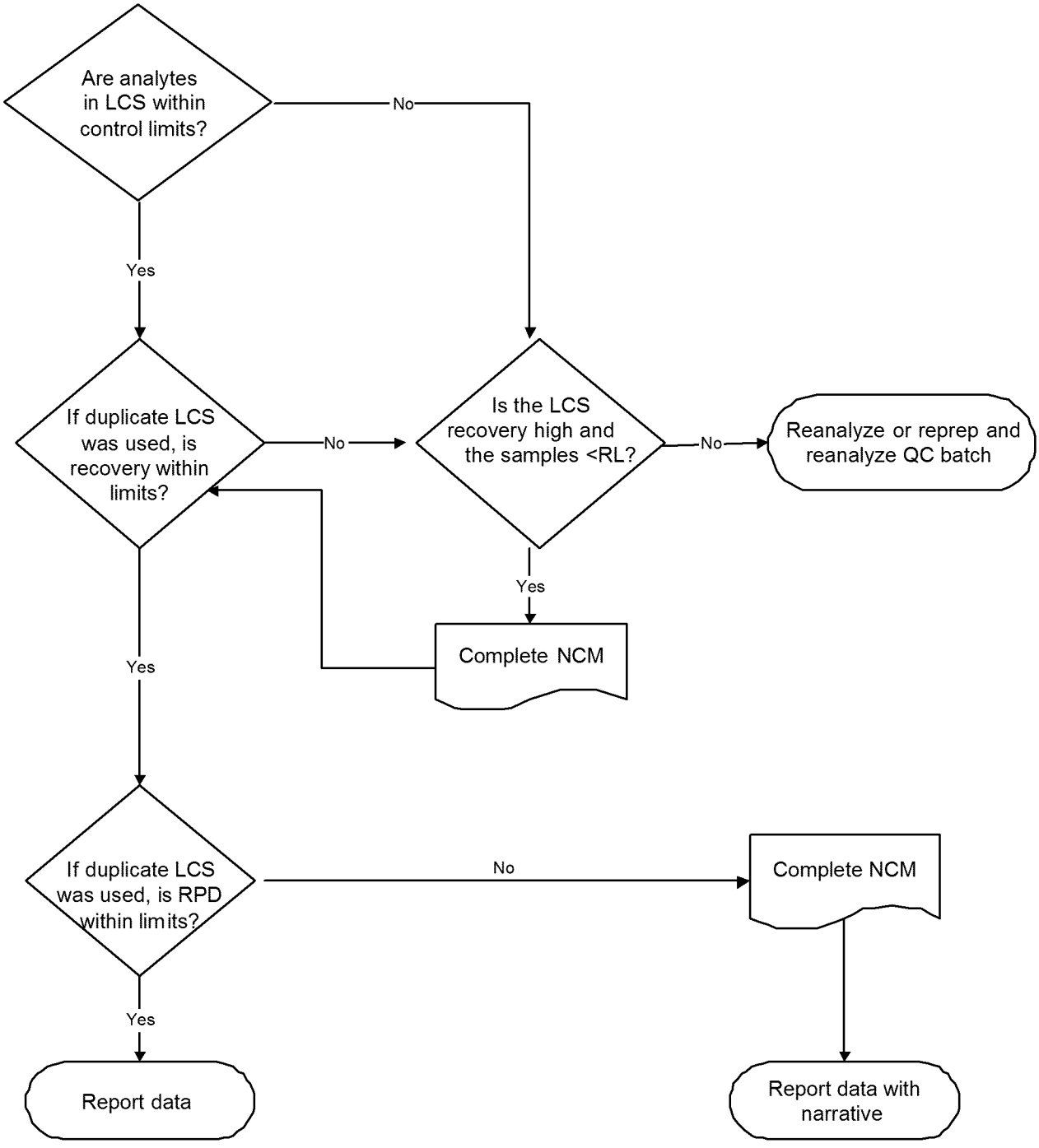


Figure 3 - Surrogate Evaluation

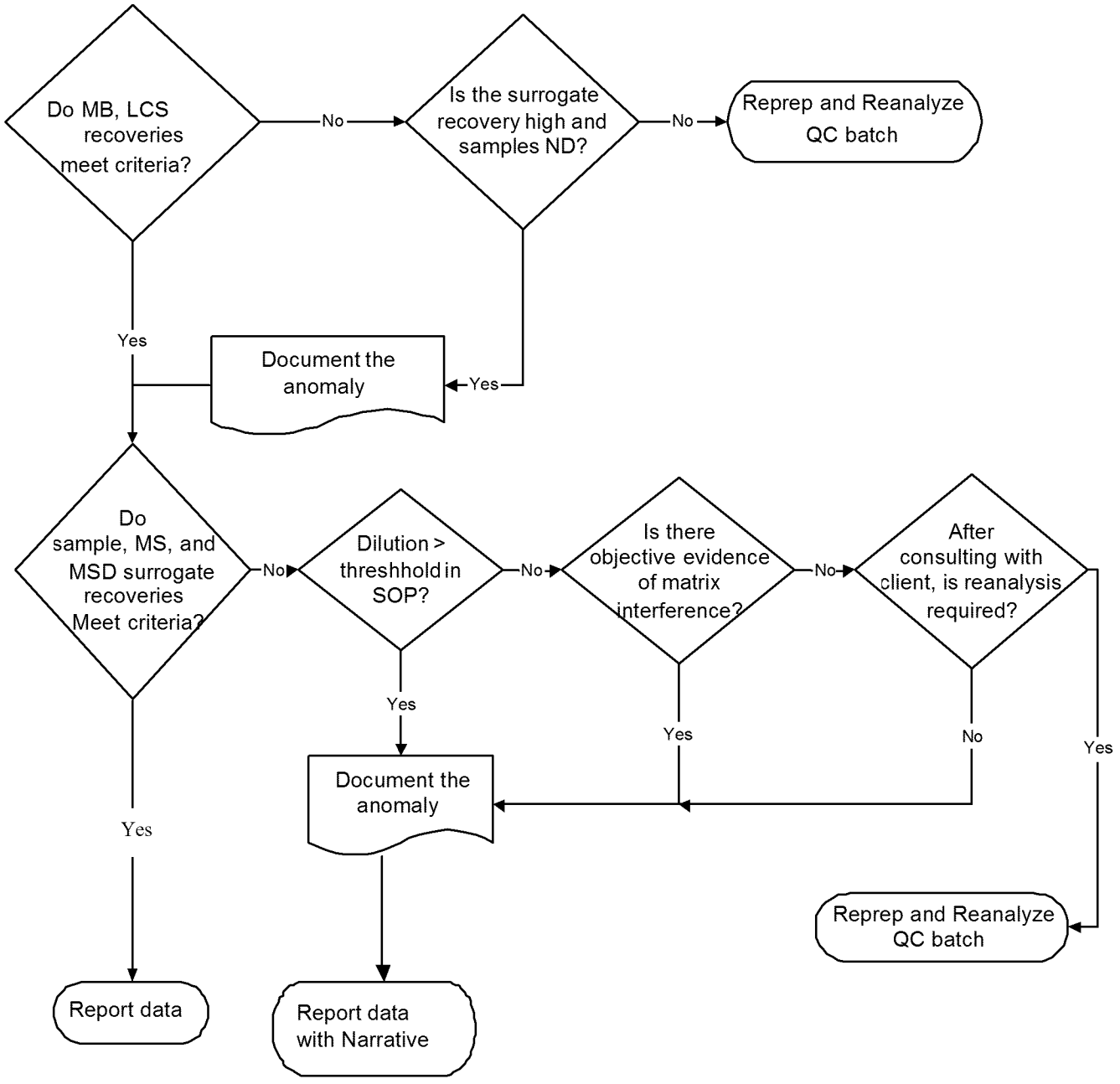
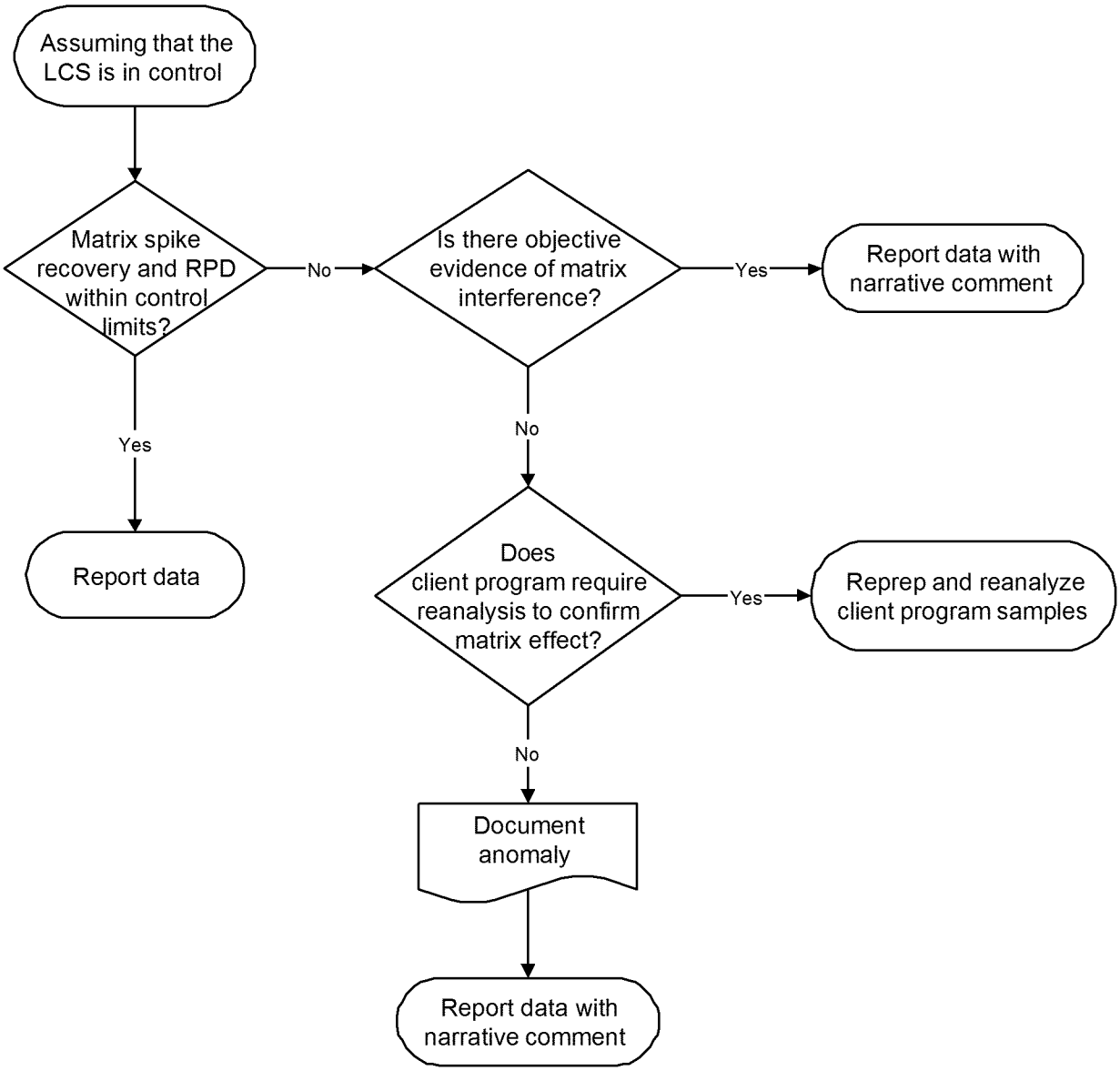


Figure 4 - Matrix Spike/Matrix Spike Duplicate Evaluation



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Canton

Policy No. QA-020, Rev. 8
Effective Date: 06/26/2013

Title: Data Validation Response and Client Complaint Handling

Approvals (Signature/Date):

Quality Assurance Manager	06/14/13 Date	Laboratory Director	06/16/13 Date
Customer Service Representative	06/12/13 Date	Operations Manager	06/12/13 Date

This SOP was previously identified as Policy No. QA-020, Rev 7 dated 11/15/11

Facility Distribution No. _____ **Distributed To: _____**

OBJECTIVE

This policy describes the TestAmerica Canton program of responding to client data validation inquiries and client complaints. Data validation inquiries and client complaints are received by the laboratory Project Manager for resolution. It is the responsibility of the Project Manager to ensure that client inquiries are responded to in a timely manner. It is the responsibility of Quality Assurance Department to oversee, provide input into data validation inquires, and track root cause analysis and corrective action activities within the laboratory. The Laboratory Director, Quality Assurance Manager, and Operations Manager are responsible for ensuring the appropriate resources are available to respond to all client validation inquiries and complaints.

SCOPE

This policy is to be enforced and followed throughout the laboratory.

RESPONSIBILITIES

QA
- Tracking Data Validations/Complaints
- Ensuring resolution to clients' and lab's satisfaction
- Overseeing Corrective Actions as required

Operations Mgt/Supervisors/ProjectManagement
- Ensuring resolution to clients' and lab's satisfaction

Operations Staff/Project Management (where applicable)
- Participating in/implementing corrective actions as required

POLICY

Data Validation

1. When the laboratory Project Manager receives data validation inquiries, s/he will:
   a. Email the client as soon as possible, confirming that inquiry is being forwarded to laboratory for review.
   b. Forward the inquiry to the associated laboratory operation group leaders, the Operations Manager, and the Canton QA distribution list. The Project Manager will specify a due date/time for return response. The collected responses will be compiled by the Project Manager – with assistance from QA Manager (or designee). The Project Manager will provide direction on whether response to the client is to be forwarded by the PM or QA. The PM will be notified as soon as possible if the requested TAT cannot be met. The Quality Assurance Manager and Operations Manager will be copied on all email traffic. The original email should include, at a minimum, the following items:

      Email subject line:
      - Start with the acronym "DV:", followed by client name & job number

Email body:

- Include job/sample numbers where applicable
- Outline the question – or include the original email from the client
- Include a due date deadline for response
- Assign responsibility for responding to client – PM or QA

2. Upon receipt of a data validation or client complaint inquiry, the laboratory Group Leader or designee must review the inquiry and the data, unless instructed otherwise. Within one business day of receipt, the laboratory must email the PM and QA with a response to the inquiry. If the due date cannot be met, the response must include an update, and a new due date that can be achieved.

3. Responses must be presented in a manner that fully explains how the error occurred and the corrective action that was taken to resolve the issue. The PM and QA must be notified immediately of any circumstances or situations that may result in the delay of a response. The Project Manager will, in turn, update the client with the status of the inquiry.

4. The Project Manager or QA (as designated in #1) will forward the client a summarized response with corrective action noted, if applicable. [In most cases, the email thread between the lab and PM should NOT be included in the response.] If the client requests further clarification or responses, the Project Manager must follow the aforementioned steps – maintaining a consistent subject line, if possible – in order to resolve the issues. When all issues have been answered to the satisfaction of the client, the Project Manager will forward the original email to QA with CLOSED added to the subject line. The Quality Assurance Department uses this date as the closure date for the data validation inquiry.

5. The Project Manager is responsible for informing the laboratory if reissued deliverables (reports, EDDs, CDs, etc.) are required. If changes to the data require recalculation, it is the responsibility of the Project Manager to request recalculation as a part of report revision, to add a revision note and revise case narrative details as required, and to verify content changes prior to delivery of revised report(s).

6. This process could reveal issues that require additional investigation or data recall. If this occurs, the procedures outlined in SOP CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall, should be reviewed and followed.

Client Complaints

1. When the laboratory Project Manager receives client complaint, s/he will:

   a. Email the client as soon as possible, confirming that the complaint is being forwarded to laboratory management for their review.
   b. Forward the information to the "Canton – Complaints" email address (see Table 1), and, where applicable, to the Operations Manager and the associated laboratory operation Group Leaders. The original email should include, at a minimum, the following items:

      Email subject line:

      - Start with "Complaint:" followed by client name & job number (if applicable)

Email body:

- Include specifics of the complaint and how it was transmitted, or the original email from client

2. The Project Manager, PM group leader and QA designee will meet within one business day of receipt to evaluate root cause(s), and whether additional investigation is needed.

3. The Quality Assurance Department is responsible for tracking all inquiries and reporting to management on a monthly basis. Evaluation for any trends or system improvements must be performed on an ongoing basis.

4. Revision History

Historical File:		Revision 0: 01/23/02		Revision 6: 10/20/10
		Revision 1: 09/17/04		Revision 7: 11/15/11
		Revision 2: 10/29/04		
		Revision 3: 08/03/07		
		Revision 4: 07/17/08		
		Revision 5: 09/18/09		

Table 1 Canton – Complaints (canton-complaints@testamericainc.com) Email Distribution List
Laboratory Director
Quality Assurance Manager/Coordinator/Specialist
Project Management Supervisor
Operations Manager

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Canton

SOP No. NC-QA-012, Rev. 3
Effective Date: 09/13/13

Title: SHIPPING DEPARTMENT

[Method: None]

Approvals (Signature/Date):

Technology Specialist	09/04/13 Date	Health & Safety Coordinator	09/06/13 Date
Quality Assurance Manager	09/13/13 Date	Laboratory Director	09/04/13 Date

This SOP was previously identified as SOP No. NC-QA-012, Rev 2.9, dated 07/09/12

Facility Distribution No. _____ **Distributed To: _____**

TABLE OF CONTENTS

1. PURPOSE

1.1. The procedures listed in this document will describe the responsibilities of Shipping Dept. personnel in ensuring that all bottle orders are sent correctly and on time.

1.2. This document accurately reflects current Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory.

2. RESPONSIBILITIES

2.1. It is the responsibility of the employee to perform the procedure described herein in full compliance with this SOP.

2.2. It is the responsibility of the Laboratory Director, QA Manager, and Departmental Supervisor of this facility to ensure that the procedures described are performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable the employee to perform the procedures in this SOP correctly.

3. SAFETY

3.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

3.2. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** Employees must review the information in the SDS for each material before using it for the first time, or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Hydrochloric Acid	Corrosive	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and

	Poison		upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions will cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Bisulfate	Irritant	None	Causes mild to severe irritation to the eyes. Prolonged exposure may cause burn if not flushed with water. May cause mild irritation to skin. Prolonged exposure may cause burn if not flushed with water.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.

Sulfuric Acid	Corrosive	None	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. May cause pulmonary edema, a medical emergency. Pulmonary edema may be delayed up to 48 hours. Contact with skin may cause redness, pain or severe pain. Contact with eyes may cause blurred vision, redness, pain, severe tissue burns or eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

3.3. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. Personal Protective Equipment (PPE) which includes lab coat, disposable gloves and safety glasses will be worn when packaging Environmental Samples. Per EHS even those with field crew and or courier clothes, must don a laboratory coat when handling environmental samples. When packaging Sampling Bottle Kits, PPE is not worn, so as to prevent sampling kit contamination, so long as preserved bottles remain unopened. Cut resistant gloves are worn under disposable gloves, only when cleaning empty coolers, which may contain shards of glassware.

3.4. All sample containers (bottles, carboys, etc.) must be securely closed to prevent leakage and placed within a secondary outer container such as a cooler.

3.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

4. SHIPPING REQUIREMENTS

4.1. Requirements for Shipping DOT Hazardous Materials and/or IATA Dangerous Goods

4.1.1. Any material meeting the definition of one of the nine DOT hazard classes (refer to Section 4.2.3) is considered a hazardous material when offered for domestic shipment by ground, rail, air, or vessel.

4.1.2. Any material meeting the definition of one of the nine IATA hazard classes (refer to Section 4.2.3) is considered a Dangerous Good when offered for transportation by air internationally. Note: A common carrier may require a domestic air shipment of a DOT hazardous material to be shipped in accordance with IATA regulations.

4.1.3. The following are the nine DOT and IATA hazard classes

4.1.3.1. Explosives (Class 1)

4.1.3.2. Compressed gases (Class 2)

4.1.3.3. Flammable liquids (Class 3)

4.1.3.4. Flammable solids, spontaneously combustible, and dangerous when wet compounds (Class 4)

4.1.3.5. Oxidizers and peroxides (Class 5)

4.1.3.6. Poisons or toxins (Class 6)

4.1.3.7. Radioactive materials (Class 7)

4.1.3.8. Corrosive materials (Class 8)

4.1.3.9. Miscellaneous materials (Class 9)

4.1.4. The following preservatives commonly added to sample bottles or vials are considered DOT hazardous materials and/or IATA dangerous goods when offered for transportation--hexane;hydrochloric acid; methanol; nitric acid; sulfuric acid, sodium bisulfate and sodium hydroxide (see Appendix IV).

4.1.5. Any sample bottle or vial containing any of the materials listed in Section 4.2.4 and being shipped or delivered to a client, Service Center, or TestAmerica Lab must be shipped in accordance with full DOT regulations unless shipped using one of the following two exceptions.

4.1.5.1. Materials of Trade per 49 CFR 173.6

4.1.5.2. Small Quantity Exceptions per 49 CFR 173.4

4.2. Materials of Trade Exception

4.2.1. Under 49 CFR 173.6, the samples that are analyzed by the laboratory are classified as a "Material of Trade." Under the provisions of 49 CFR 173.6,

"Materials of Trade" are not subject to the provisions of the hazardous materials shipping regulations as long as the following provisions are met.

4.2.1.1. The material is transported by TestAmerica employees, the client's employees, or private courier hired by TestAmerica or the client.

4.2.1.2. The total gross aggregate weight of the sample package does not exceed the limits set forth in the citation. Individuals need to check the regulatory citation since the total mass varies by hazard class and packing group.

4.2.1.3. The total gross aggregate weight of all packages containing known hazardous materials does not exceed 440 pounds.

4.2.1.4. The materials are packaged in accordance with the citation. Packaging for each classification of material may vary slightly. However, in general the packages must be leak tight for liquids and gases, sift-proof for solids, securely closed, secured against movement, and protected against damage.

4.2.1.5. The outer packages are marked with either a common name (example: Hexane) or a proper shipping name (example: sulfuric acid mixture).

4.2.1.6. The operator of a motor vehicle that contains a material of trade must be informed of the presence of the hazardous material.

4.2.2. When using this exception, there are limitations on the quantity of hazardous materials permitted in individual containers for the various hazard classes. Contact the EHSC or Shipping Manager prior to using this exception.

4.3. Small Quantity Exception

4.3.1. Under 49 CFR 173.4, sample kits containing hazardous materials listed in Section 4.2.4 are not subject to the provisions of the DOT hazardous materials shipping regulations or IATA dangerous goods regulations as long as the following provisions are met:

4.3.1.1. The amount of material in each inner package may not exceed 30 ml or 30 g.

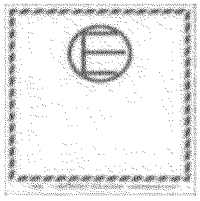
4.3.1.2. The inner package must either be plastic having a minimum thickness of no less than 0.2 millimeters (0.008 in), or earthenware, glass or metal.

4.3.1.3. The inner package must be packed with a secure material that will not react with the material in the container and will absorb all liquid present.

4.3.1.4. The inner packages must be packed in a strong outer package that can withstand the Drop and Stack tests specified in the citation (refer to Section 4.6.5). To summarize these tests, the package must be able to be dropped from 5.9 ft. on any corner or side without any containers breaking or leaking and must be able to withstand being stacked to a height of ten feet for 24 hours without collapsing.

4.3.1.5. The total gross aggregate weight of each package may not exceed 64 pounds.

4.3.1.6. If hazardous materials are shipped under the provisions of 49 CFR 173.4, the following statement, "This package conforms to 49 CFR 173.4 for domestic highway or rail transport ONLY", must be included on the outside of the package when shipped ground (see Appendix V).



4.3.1.7. If the sample kits are shipped under the provisions of the IATA regulations, a "Dangerous Goods in Exempt Small Quantity" label must be completed and attached. Sample kits shipped internationally must be shipped under the IATA provisions.

4.3.1.8. Nitric Acid in concentrations of 20% or less may be shipped by air under the IATA provisions. The maximum amount of material in the outer packages may not exceed 500ml. The inner package must not exceed 30 ml.

4.3.1.9. Nitric acid is not permitted to be shipped via air using the "Dangerous Goods in Exempt Quantities" provision.

4.3.1.10. Nitric Acid in concentrations greater than 20% is forbidden to be shipped via air using the 49 CFR 173.4 provisions. Shipment of this material must be made by ground only.

4.4. Requirements for Shipping Known Samples of Hazardous Waste

4.4.1. Under the provisions of 40 CFR 261.4(d), samples are excluded as "Hazardous Waste" as long as they meet the following requirements.

4.4.1.1. The sample is being transported to a laboratory for the purpose of testing.

4.4.1.2. The sample is being transported back to the sample collector after testing.

4.4.1.3. The sample is being stored by the sample collector prior to transportation to a laboratory for testing.

4.4.1.4. The sample is being stored in a laboratory before testing.

4.4.1.5. The sample is being stored in a laboratory after testing but before it is returned to the sample collector.

4.4.1.6. The sample is being stored temporarily in the laboratory after testing for a specific purpose (for example, until conclusion of a court case or enforcement action, where further testing of the sample may be necessary).

4.4.2. As long as the sample does not meet one of the other definitions of a "Hazardous Material" under DOT regulations it is not a DOT "Hazardous Material".

5. PROCEDURES

5.1. Any deviations from this procedure must be documented as a nonconformance with a cause and corrective action described.

5.2. Sample Bottles Filled by Clients or TestAmerica Associates

5.2.1. As mentioned in Section 4.2.4, the seven DOT "Hazardous Materials" that are used as preservatives are Hexane, Nitric Acid, Sulfuric Acid, Hydrochloric Acid, Sodium Hydroxide, Sodium Bisulfate and Methanol.

5.2.2. With regard to the acids and bases that are used to preserve samples, as long as the amount of preservative in the original empty sample container does not exceed the amount listed in Appendix I, the sample will not meet the definition of a DOT corrosive material when filled with water by the client or an TestAmerica associate, and may be shipped as a non-hazardous material to laboratories for analysis.

5.2.3. Samples preserved with Methanol and Sodium Bisulfate and wipe samples preserved with Hexane are still DOT "Hazardous Materials". They may be shipped under the provisions for hazardous materials in excepted quantities as long as there is less than 30 ml per container being returned to the lab. The volume in the container does not include soil added to the vials with the Methanol. However, it does include the volume of water added to the vials containing Sodium Bisulfate.

5.2.4. Preservatives not listed in Appendix I shall not be used unless approved by the EHSC/EHSD.

5.2.5. Drop and Stack Test

5.2.5.1. Coolers or foam boxes containing glass-preserved bottles must be lined with four layers of ½" bubble wrap. One-liter glass-preserved bottles must to be in a 5/16" bubble bag. Air pillows are used to fill all open areas and dead space. Layers of bubble wrap are placed between the top of the cooler and the lid. The following types of coolers are used:

Small foamer
Large foamer
28-quart cooler
50-quart cooler
68-quart cooler

5.2.5.2. Stack Test – Stack the coolers, and let them sit for at least 24 hours. The stack must be at least ten feet tall. Take a digital photo of the coolers at the beginning and end of the test period. A negative test would be indicated if any of the coolers collapsed during this test period.

5.2.5.3. Drop Test – Each type of cooler listed in Section 4.6.5.1 is dropped from a height of 5.9 ft. onto a solid unyielding surface. Coolers are dropped in each of the following orientations:

Flat on the bottom
Flat on the top
Flat on the long side
Flat on the short side
On a corner at the junction of three intersecting edges

A different cooler is used for each drop (i.e., five different coolers are used for each cooler type—one for each drop.) After each cooler is dropped, a digital photo is taken of the contents. A negative test is indicated if any of the containers break, material leakage from the inner container occurs, or a substantial

reduction in effectiveness of the cooler is observed. If any of the coolers fail the test, the procedures for packaging the coolers must be reviewed and changed accordingly. The drop tests must then be repeated.

5.2.5.4. Drop Test and Stack Test results conducted at the lab are documented in a separate report. The lab had 100% "no breakage" for each test.

5.3. Creation of LIMS Shipping Order

5.3.1. The project manager will post a Shipping Order onto the Shipping Desktop in LIMS. Status of Shipping Order when posted is "Ready To Process", and Shipping Order shows up as highlighted green in color on the desktop.

5.4. Receiving Shipping Orders from Project Management

5.4.1. The shipping staff will monitor the Shipping Desktop throughout the course of the workday, refreshing the desktop periodically. New, recently posted Ready to Process shipping orders will show up green.

5.4.2. The shipping staff will click on green "Ready to Process" shipping orders. Upon selection of orders, staff can then elect to print 1) A hardcopy of the Shipping Order Form, 2) Chain of Custody, and/or 3) Bottle Labels. Both Chain of Custody and Bottle Labels will print with the client sampling identifications previously generated.

5.4.3. Upon electing to print, shipping order then moves to "In Process" status. Orders "In Process" are highlighted as yellow on the shipping desktop.

5.4.4. The shipping staff will then review the Shipping Order Form for accuracy of selections made by Project Management. Orders are prioritized by due date and size (based upon current bottle inventory).

5.4.5. Shipping Orders include 1) Client and Project description, 2) Client Address for shipment, 3) Due date, 4) Level of Expedience and 5) Manager of Project. Also included are 1) Number of Sets, 2) Bottles per Set, 3) Bottle Type Description, 4) Preservative, 5) Method, 6) Matrix, 7) Sample Type, 8) Comments to Field Samplers and Shipping Department.

5.5. Packaging of the Shipping Order

5.5.1. The shipping staff will pull requested bottles from inventory and begin to set up according to selections of project management noted on the Shipping Order Form.

5.5.2. Depending upon the matrix and requested bottle preservation, shipping staff will package bottleware per Department of Transportation (DOT) regulations.

5.5.3. Staff will package bottles and/or supplies into hard-side coolers or Styrofoam coolers, depending upon client request. Clients may also request that only cases and/or bottles be shipped in cardboard boxes. Occasionally clients will also request arrangement of a Drop Ship of supplies directly from bottle vendor to client.

5.5.4. As containers are pulled from the inventory and subsequently packaged into coolers, two important numbers are recorded on the shipping order form. First, the lot number of the containers Production Date is noted. Second, the lot number of the Acid Batch is noted (if preserved). Vendor name is also notated upon the lot tag.

5.5.5. If preserved bottles are needed, prepare in the following order:

5.5.5.1. Line bottom of cooler with absorbent padding

5.5.5.2. Place four layers of ½ inch bubble wrap

5.5.5.3. Heavy plastic liner, opened

5.5.5.4. Inside liner, place absorbent pad

5.5.5.5. Place sample bottles on top of pad, keeping them upright

5.5.5.6. Twist liner closed over sample bottles

5.5.6. If unpreserved containers are needed, prepare in the following order:

5.5.6.1. Line bottom of cooler with heavy plastic liner, opened

5.5.6.2. Place containers on liner, and twist liner closed over containers

5.5.7. Upon completion of cooler packaging, paperwork is included in the main cooler of a set. Shipping paperwork includes at minimum; Chains of Custody, Labels, Custody Seals, Department of Transportation (DOT) Regulation Compliance Flyer, sampling and bottling instructions, and a copy of the Shipping Order Form.

5.5.8. Labels are applied to the rear exterior of the cooler. Applicable labels include at a minimum; Small Quantities Exceptions, Fragile Glass and This Side Up. Main cooler is clearly delineated as containing shipping paperwork If cooler is part of a set, it is noted as well on the Sampling Instructions Enclosed label (1 of 5 for example). After the container is strapped or taped closed, Shipping Dept.

personnel will place an address label on the shipping container. If there are preservatives inside the container, hazardous labels will be applied. The label must read, "This package conforms to 49 CFR-173.4 for domestic highway or rail transport ONLY" if being shipped by ground. If being shipped by air, the "E" label is to be placed on the outside of the container with the proper hazard class under the "E", along with the address of the shipper if not already on the package (see Appendix III for hazard classes). A chemical emergency contact label must also be present.

NOTE: Custody seals will be used by client request.

5.5.9. Packed coolers are sealed tightly using a mechanical strapping machine. At a minimum, 2 bands of strapping are applied. The strap is initialed and dated using a permanent marker. Finally a heavy paper luggage tag is adhered to the cooler.

5.5.10. The maximum volume of preservative in each container shall not exceed the values listed in the following table. See Appendix I for maximum volumes.

5.5.11. Parts of the above table were derived from the table listed in Section 4.6.3. The sample container sizes and maximum volumes are based on the sample container sizes used at TestAmerica Canton.

5.5.12. Upon client request, one trip blank is shipped with each cooler requiring volatile analysis. Trip blanks can be supplied for other test methods if requested by the client.

5.5.13. Equipment and field blanks are also supplied, if requested.

5.5.14. All sample containers (bottles, carboys, etc.) must be securely closed to prevent leakage and placed within a secondary outer container such as a cooler.

5.6. Shipping of Packaged Coolers

5.6.1. Packed coolers are placed on the scale of the Federal Express Shipping Manager (FESM) computer terminal. For sets of coolers, one cooler representing the set is weighed. The weight is uploaded to the (FESM) shipping data screen. Other relevant shipping information is entered into the (FESM), such as 1) Client Name 2) Client Recipient 3) Client Address and Telephone 4) Number of coolers in set and 5) Method of expedience.

5.6.1.1. Method of expedience should be determined by how soon the coolers are needed, cooler contents, and shipping distance.

5.6.2. After selecting the method of expedience, an adhesive airbill is printed and placed upon the cooler luggage tag. Coolers are then stacked on large

pushcarts (maximum capacity of 9 large coolers) and staged in the shipping warehouse, awaiting pickup.

5.7. Documentation

5.7.1. After the order is completed, charges (if any) will be logged in LIMS.

5.7.2. Bottle and preservative lot numbers are recorded on the bottle chain of custody.

5.8. Return of Coolers

5.8.1. After coolers are unpacked in Sample Receiving, they are brought to Shipping. The cooler receipt is documented on log sheets and entered in the computer system as received.

5.8.2. The inner plastic liner is disposed of and the cooler is cleaned and stored for additional usage.

5.9. Packaging of Environmental Samples for Workshare

5.9.1. Depending upon workload of environmental sample receipt and the laboratory capacity, samples may be shipped to other facilities.

5.9.2. Project Management will coordinate with the importing facility to export environmental samples. Mangers of projects will verify that importing facility can 1) Qualify certification 2) Run analysis and 3) Meet turn around times. Managers of projects will advise Sample Receiving staff as to the destination of samples being exported via Workshare. Receiving will print from LIMS Shipping Desktop an Interlab Chain of Custody (ICOC).

5.9.3. Receiving staff will place samples destined for Workshare exporting on a cart. Prior to close of business, receiving staff will communicate with shipping staff regarding what samples will go where. Samples will include the appropriate (ICOC), which will designate samples by Job Number, Analysis and Destination.

5.9.4. The shipping staff will package one shipment at a time. While packaging samples shipping staff will the check the bottle label to confirm samples are in the correct shipment.

5.9.5. Coolers are used when shipping environmental samples for Workshare and are prepared as follows:

5.9.5.1. Layers of bubble wrap are placed upon the inside bottom of cooler.

5.9.5.2. A heavy liner is opened and placed on top of the bubble wrap.

5.9.5.3. Glass bottles are individually wrapped in bubble wrap. Plastic bottles do not require bubble wrap.

5.9.6. Bottles are placed in an upright position. If coolant is needed, the bottles are covered in ice. At least 10-20 pounds of ice coolant is added to samples. The staff makes an attempt to limit total cooler weight (samples and coolant) to no more than 60 pounds. If total weight is heavier, then samples are divided between two coolers.

5.9.7. After adding proper coolant, the staff includes a temperature blank and then uses a zip tie to close the poly liner. A copy of the (ICOC) paperwork is placed inside cooler on top of closed liner bag.

5.9.8. Sample cooler is then labeled with "Wet Ice" and either "Fragile" or "Glass" stickers as well as "This Side Up" arrows. If sample coolers are packed into sets, one cooler of the set must include ICOC and be labeled as Sampling Instructions Enclosed.

5.9.9. Sample cooler is then mechanically sealed as note in section 5.5.9.

6. DEFINITIONS

6.1. Refer to glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current version.

7. POLLUTION PREVENTION

7.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

8. WASTE MANAGEMENT

8.1. Waste Management

8.1.1. All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

8.1.1.1. Waste Streams Produced by the Method

8.1.1.1.1. Preservatives received in client-returned bottles. This waste is disposed of in a designated container identified as "Acid Waste".

9. REFERENCES

9.1. References

9.1.1. TestAmerica Canton Quality Assurance Manual (QAM), current version

9.1.2. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

9.1.3. Corporate Quality Management Plan (CQMP), current version

9.1.4. WI-NC-047A Hot Weather Sample Packing Instructions, current version

9.1.5. WI-NC-050A Low-Level Hg Sample Collection, current version

9.1.6. WI-NC-053A Sample Packing Instructions, current version

9.1.7. WI-NC-056A Aqueous VOC & TOC Instructions, current version

9.1.8. WI-NC-059A Low Level Field Preservation Sample Kit Instructions, current version

9.1.9. WI-NC-061A Wipe Sampling Procedure, current version

9.1.10. WI-NC-064A General Sample Bottle Kit Instructions, current version

9.1.11. WI-NC-067A Solid VOC Instructions, current version

9.1.12. WI-NC-070A MEOH Field Preservation Kit Instructions, current version

9.1.13. Memo from Nate Nunn, TestAmerica Inc. EHS Director, to EHSCs, GMs, LDs, K.Wheatstone, and C.Carter, Subject: Shipments of Samples, dated 4/17/02

9.1.14. TestAmerica Canton 2007 Drop Test and Stack Test

9.1.15. Revision History

Historical File:		Revision 2.0: 04/27/99		Revision 2.7: 12/30/09
		Revision 2.1: 06/30/03		Revision 2.8: 04/28/11
		Revision 2.2: 08/30/06		
		Revision 2.3: 12/06/06		
		Revision 2.4: 07/10/07		
		Revision 2.5: 05/20/08		
		Revision 2.6: 06/18/09		

10. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

10.1. Appendix I: Preservative Maximum Volume Table

10.2. Appendix II: Hazard Class Labels

10.3. Appendix III: Regulatory Compliance Notice

Appendix I: Preservative Maximum Volume Table

Preservative	Sample Container Size	Maximum Volume	Hazard Class	Shipping Mode Allowed for Containers Being Shipped
Hexane	60 ml wide mouth 40 ml VOA vial	30 ml 30 ml	3	Air or Ground
Methanol	60 ml wide mouth 40 ml VOA vial	30 ml 30 ml	3	Air or Ground
Sodium Bisulfate	40 ml VOA vial	30 ml	8	Air or Ground
Sulfuric Acid: 1:4 Concentrated acid in water Sulfuric acid 1:1 Concentrated acid in water	250 ml 500 ml 1 Liter40 ml VOA vial	2 ml 4 ml 8 ml 0.5 ml	8	Air or Ground
Nitric Acid 1:1 Concentrated acid in water	1 Liter	6 ml	8	Ground only
Nitric Acid: 1:4 Concentrated acid in water	250 ml 500 ml 1 Liter	2 ml 4 ml 8 ml	8	Air or Ground
Hydrochloric Acid 1:1 concentrated acid in water Hydrochloric Acid 1:1 concentrated acid in water	40 ml VOA vial 1 Liter	0.2 ml 5 ml	8	Air or Ground
Sodium Hydroxide 4N	250 ml 500 ml	2 ml 3 ml	8	Air or Ground
Sodium Hydroxide 10N	500 ml	1.25 ml	8	Air or Ground

Appendix II: Example of Hazard Class Labels

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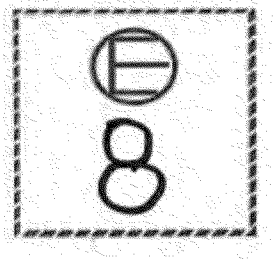
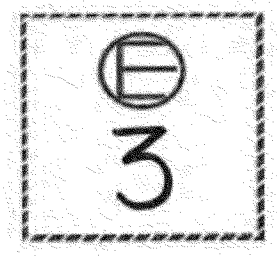
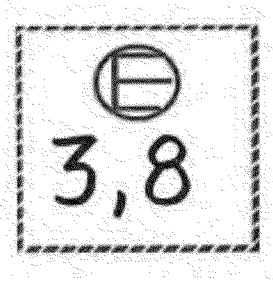


For Corrosives only (Hydrochloric Acid, Sulfuric Acid, Nitric Acid, Sodium Hydroxide, Sodium Bisulfate)



For Flammable liquids only (Methanol, Hexane, Isopropanol)



For Combination of corrosives and flammable liquids or when shipping Teracores

ENSURE TESTAMERICA MOBILE'S NAME AND ADDRESS ARE SOMEWHERE ON COOLER AS WELL AS NAME AND ADDRESS OF CLIENT

ENSURE TO FOLLOW PRESERVATIVE AMOUNTS IN BOTTLE PREP

Appendix III: Regulatory Compliance Notice

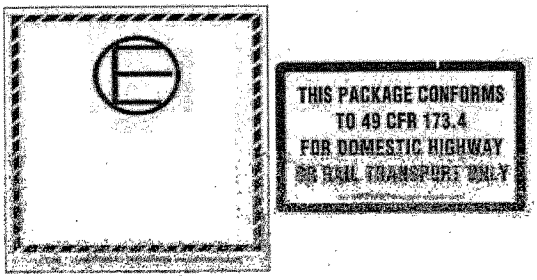
REGULATORY COMPLIANCE NOTICE !

Important Instructions for Shipping Samples to TestAmerica Laboratories.

Option #1 –all bottles being shipped contain samples (no unfilled containers).

Prior to shipping samples to TestAmerica Laboratories, the shipper must be sure the hazardous-material markings and the associated MSDS forms match the contents of the shipment. If markings are incorrect, shipments may be delayed by commercial carriers.

When all bottles contain samples (no unfilled containers), remove the hazardous-materials labels (examples shown below) from the outside of coolers and remove all Material Safety Data Sheets from the packet provided in the cooler.



Option #2 – some bottles being shipped are unfilled pre-preserved containers.

When unused pre-preserved containers cannot be discarded, and are being shipped to TestAmerica, leave hazardous-materials markings (as shown above) on the outside of the cooler.

Apply the label "This container also contains non regulated environmental samples."

MSDS's may be shipped back with the cooler or discarded.

Remember it is the shipper's responsibility to be compliant.

Page 2 contains additional guidance on DOT & IATA compliance.

NOTE: These advisory documents are not intended to be used in lieu of official regulatory compliance instructions but rather as a guide. It is the shipper's responsibility to follow and interpret all applicable DOT and IATA regulations.

Shipping Instructions to Comply with Department of Transportation (DOT) & International Air Transport Association (IATA) requirements

Returning Samples to TestAmerica

When a pre-preserved bottle is filled with sample, it is no longer considered a DOT excepted hazardous material. **Before shipping environmental samples back to the laboratory, please remove all DOT labels from the outside of the cooler.** Two common label formats are indicated below:

- **Any form of the label** 
- **"This package conforms to 49CFR 173.4 for domestic highway or rail transport only"**

Note: If one or more of the samples being shipped to the laboratory is known to be a hazardous material, or if the client suspects that a sample meets one of the classifications of a hazardous material, then it needs to be shipped in accordance with 49 CFR 172.101(c)(11).

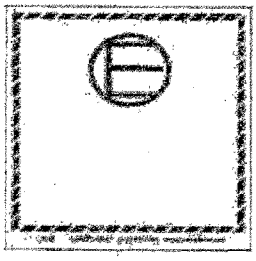
Returning Unused Bottles to TestAmerica

DOT labeling must be used if you are returning unused pre-preserved bottles to the lab. (Unpreserved bottles have no special labeling requirements.) It is best to pack unused bottles in a separate cooler from environmental samples.

For shipment via ground transportation, cooler/box needs to bear the label "This package conforms to 49CFR 173.4 for domestic Highway or rail transport only."

For shipment via air transport, cooler/box needs to bear the label "E." The hazard class (i.e., 3 or 8) must be marked under the "E" (see examples below), and the name, address & emergency phone number of the shipper or consignee must be marked on the outside of the cooler/box.

Examples:



For Corrosives only (Hydrochloric Acid, Sulfuric Acid, Nitric Acid, Sodium Hydroxide, Sodium Bisulfate)

For Flammable Liquids only (Methanol, Hexane, Isopropanol)

For Combination of corrosives and flammable liquids [Including TerraCore kits w/vials containing Sodium Bisulfate (8) and Methanol (3)]

If you have any questions or need additional information, please call your Service Center or Project Manager, or contact your commercial carrier directly.

NOTE: These advisory documents are not intended to be used in lieu of official regulatory compliance instructions but rather as a guide. It is the shipper's responsibility to follow and interpret all applicable DOT & IATA regulations.

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TestAmerica Canton

SOP No. NC-QA-013, Rev. 1.7
Effective Date: 08/20/12

Title: INVENTORY/WAREHOUSE CONTROL

[Method: None]

Approvals (Signature/Date):

[redacted]	08/20/12	[redacted]	07/17/12
Technology Specialist	Date	Health & Safety Coordinator	Date
[redacted]	08/19/12	[redacted]	07/17/12
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP NC-QA-013, Rev 1.6, dated 05/31/11

Facility Distribution No. _____ **Distributed To: _____**

TABLE OF CONTENTS

1. PURPOSE

1.1. The procedures listed in this document describe the responsibilities of Warehouse Management and Inventory Control personnel in ensuring that all laboratory and office equipment and supplies are obtained cost effective and maintained efficiently.

1.2. This document accurately reflects current Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory.

2. RESPONSIBILITIES

2.1. It is the responsibility of the employee to perform the procedure described herein in full compliance with this SOP.

2.2. It is the responsibility of the Laboratory Director, QA Manager, and Departmental Supervisor of this facility to assure the analysis described is performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable the employee to perform this SOP correctly.

3. SAFETY

3.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

3.2. Normal office-dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.

3.3. The Warehouse Manager will take a forklift training course every three years, and will comply with all safety rules when operating the forklift.

4. PROCEDURES

4.1. Any deviations from this procedure must be documented as a nonconformance with a cause and corrective action described.

4.2. On a daily basis:

4.2.1. In J.D.Edwards – receive packing slips weekly.

4.2.1.1. Select – receive by P.O.

4.2.1.2. In "Order Number", type the P.O. number of items received. Then click "Find." The order will appear on the screen. Select the appropriate row, and click "Select" in the "Rec Opt" column.

4.2.1.3. In the "Rec Opt" box, enter "1". In the "Quantity" column, enter the amount of the item(s) shipped on the packing slip; then click "OK". This is standard on all purchase orders received. Once the remaining items come in, follow the steps in Section 4.2.1.2 and 4.2.1.3.

4.2.1.4. All packing slips will be entered daily, and an Open Purchase Order report will be run once per month.

4.2.2. Check consignment areas to make sure supplies are not low. If supplies are low, call the designated representative through Fisher, Agilent, or Restek; and give them the catalog number of item(s) needed.

4.2.3. Check the dock periodically throughout the day, and deliver any packages received to the correct recipient(s).

4.2.4. Check the stock of paper and folders. Order through Staples when low.

4.2.4.1. On-line at Staples.com

4.2.4.2. Enter user ID and password

4.2.5. Ethyl ether is ordered through MG Scientific. Pipettes are ordered through Rainin and Gilson. Cyclotainers are ordered through MG Scientific. Mason Jars are ordered through MG Scientific. Amber-coated bottles are ordered through Qorpak. These are all ordered in J.D.Edwards.

4.3. Monday, or as Needed

4.3.1. Staples order – supplies such as copier paper, printer and fax supplies, folders, etc. All other toners are ordered through Copeco.

4.4. Friday before Noon

4.4.1. Air Product order – request PO number; then fill out Air Product Order Form.

4.4.1.1. The following quantity of each gas is on-site at all times:

Upstairs:
Air - 3
Helium – 6

Argon - 3
Nitrogen - 6
Oxygen – 2
Hydrogen – 4
NI CZ 300- 4

Downstairs:
Helium - 8
Nitrogen – 8
Air - 2

4.4.1.2. For example, if there are only four Helium downstairs, order four to bring the total back up to eight.

Note: Bulk Argon is filled on a scheduled weekly basis. Once filled, a requisition will be entered into JDE. The cost breakdown is as follows: total SCF used divided by 100 x $3.41. This will break down the cost per fill-up. Bulk Nitrogen is scheduled on a weekly basis. Breakdown is the same as Argon, but at $1.15 SCF.

4.4.1.3. Fill in the order sheet, including the order number and date; then enter order into J.D.Edwards. Upon approval from the Ops Manager, Purchasing will enter the PO number on the order form and fax it to Air Gas Products.

4.5. Tuesday and Thursday

4.5.1. Scan out all products taken from the consignment area, and do the download. This will generate an order that is usually in by the next day.

4.6. How to scan and download signed-out products in consignment area:

4.6.1. Remove scanner from the base.

4.6.2. In the main menu field, click on "Disbursement"

4.6.3. "Enter or Scan Badge" appears.

4.6.3.1. Scan the group leader's bar code on the folder. It will automatically default to the next screen

4.6.3.2. "Cost Center/Value 1" will appear. Scan the department bar code below the group leader's bar code.

4.6.3.3. Click on "Accept"

4.6.4. Entering Stock Numbers.

4.6.4.1. Scan the 1st item from the sign out sheet from that department.

4.6.4.2. The part number will appear in the "stock number" box. Verify the number is correct.

4.6.4.3. The system will default to the next field, which is "Quantity". Enter the amount signed out, and units (ea, pk, cs, etc.).

4.6.4.4. Chose "More" if there are more items to sign out from that group.

4.6.4.5. Once all items are scanned from that department, click "Enter". This will take you back to the main menu.

4.6.4.6. Repeat steps 4.6.2 through 4.6.4.5 for other departments.

4.6.5. Once all folders have been scanned out, place the scanner back in the base.

4.6.6. Active Sync will connect automatically on the desktop. Once connected, click on the MC55 Desktop icon.

4.6.6.1. Click on "Move Data From Scanner"

4.6.6.2. Click on "Upload Disbursements". "Test or Production" screen will appear. Chose "Production" and wait for "uploads" to upload scanner

4.6.6.3. When the upload is finished, a green screen reading "Transmission successful" will appear. Click the red "X" box three times, and then click the red "X" box on Active Sync.

4.6.6.4. Send an email to the Fisher representative stating, "Download is in and ready".

4.6.6.5 Agilent consignment inventory is done on a monthly basis. The consignment sheet will be sent to the Agilent representative, and they will do a breakdown spreadsheet by department and send back. A requisition will then be entered into J.D.Edwards, and a P.O. number will be sent to Agilent for replenishment of supplies.

4.6.6.6 Restek consignment inventory will be done on a monthly basis. A Restek representative will come in to do an inventory count with the Warehouse Manager. The Restek representative will then send a spreadsheet, and a requistion will be made in J.D.Edwards. A P.O. number will be sent to Restek for replenishment of supplies.

4.6.7. QEC Order

4.6.7.1. The Shipping Department orders the bottles, but it is the responsibility of both the Warehouse Manager .and the Shipping Dept to put supplies away.

NOTE: While putting away any order, check quantity of products received with the packing slip and check for any discrepancies.

4.6.8. Consignment Inventory

4.6.8.1. A representative from Fisher Scientific and the Warehouse Manager perform a monthly physical inventory of consigned laboratory supplies.

4.7. Warehouse Management

4.7.1. Ensure warehouse is kept clean and organized. If any item(s) are to be placed in the warehouse for storage, the item(s) must be placed into assigned locations. Check with the Warehouse Manager for proper storage.

4.7.2. Provide adequate floor space for each department's needs

4.7.3. Maintain storage space for leased items, unused laboratory equipment, and un-consigned laboratory supplies

4.8. Assist laboratory personnel in locating and/or ordering supplies.

4.9. Pursue cost reduction strategies.

4.10. Provide backup to Shipping Department.

5. POLLUTION PREVENTION

5.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

6. WASTE MANAGEMENT

6.1. There are no waste streams associated with this procedure.

7. DEFINITIONS

7.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current version.

8. REFERENCES

8.1. References

8.1.1. Corporate Quality Management Plan (CQMP), current version

8.1.2. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

8.1.3. TestAmerica Canton Quality Assurance Manual (QAM), current version

8.1.4. Revision History

Historical File:		Revision 1.0: 03/19/01		Revision 1.6: 05/31/11
		Revision 1.1: 12/03/04		
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		Revision 1.3: 04/10/08		
		Revision 1.4: 04/09/09		
		Revision 1.5: 05/25/10		

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Canton

SOP No. NC-QA-014, Rev. 10
Effective Date: 07/12/12

Title: GLASSWARE WASHING

[Method: None]

Approvals (Signature/Date):

[redacted]	07/11/12	[redacted]	07/10/12
Technology Specialist	Date	Technology Specialist	Date
[redacted]	07/10/12	[redacted]	07/10/12
Technology Specialist	Date	Technology Specialist	Date
[redacted]	07/10/12	[redacted]	07/12/12
Technology Specialist	Date	Health & Safety Coordinator	Date
[redacted]	07/12/12	[redacted]	07/10/12
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP NC-QA-014, Rev 9, dated 04/28/10

Facility Distribution No. _____ **Distributed To: _____**

TABLE OF CONTENTS

1. PURPOSE

1.1. The procedure listed in this document will describe the standard operating procedures for washing glassware in the laboratories.

1.2. This document accurately reflects current Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory.

2. RESPONSIBILITIES

2.1. It is the responsibility of the analyst to perform the analysis described herein in full compliance with this SOP.

2.2. It is the responsibility of the Laboratory Director, QA Manager, and departmental Supervisor of the facility to assure that the analysis described is performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable the analyst to perform this SOP correctly.

3. SAFETY

3.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

3.2. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	OSHA Exposure Limit (2)	Signs and Symptoms of Exposure/Unusual Hazards
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	OSHA Exposure Limit (2)	Signs and Symptoms of Exposure/Unusual Hazards
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

3.3. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves **MUST** be worn when washing glassware. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

3.4. Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. Solvent and waste containers will be kept closed unless transfers are being made.

3.5. The glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operation will permit.

3.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Canton associate. The situation must be reported immediately to the EH&S Coordinator and to a Laboratory Supervisor.

3.7. Glassware in contact with chemicals used in analytical procedures may be toxic or carcinogenic. Therefore, each piece of glassware should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals should be reduced to the lowest possible level.

3.8. Hands must not be placed in the glassware while washing. An appropriate scrub brush must be used to clean the inside of glassware. This will prevent the breakage of glassware while trying to force hands in or out of apparatus.

4. PROCEDURES

4.1. One-time procedural variations are allowed only if deemed necessary in the professional judgement of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a NonconformanceMemo and is reviewed by a Technical Specialist. The NonconformanceMemo shall be filed in the project file.

4.2. Any deviations from this procedure must be documented as a nonconformance with a cause and corrective action described.

4.3. Metals

4.3.1. Dirty glassware is taken to a central location and thoroughly rinsed. Any ink on the outside of the glassware is removed with acetone.

4.3.2. The glassware is immersed in a hot, soapy solution of water and laboratory detergent. An appropriate scrub brush or pad is used to scrub the glassware.

4.3.3. Rinse the glassware thoroughly three times with hot tap water and then rinse once with 1:1 nitric acid. Appropriate protective wear should be worn. If the glassware is still visibly dirty, or if spotting or beading occurs, repeat Section 4.3.2.

4.3.4. Rinse the glassware three times with analyte-free water, and place it in a clean drying area.

4.3.5. After air drying, the glassware should be spot- and stain-free. If not, then repeat the entire procedure. Seldom used items should be stored to minimize contamination.

4.4. Wet Chemistry

4.4.1. Dirty glassware is taken to a central location and thoroughly rinsed. Any ink on the outside of the glassware is removed with acetone.

4.4.2. The glassware is immersed in a hot, soapy solution of water and laboratory detergent. An appropriate scrub brush or pad is used to scrub the glassware. A mechanical, laboratory dishwasher equipped with a DI water rinse is also approved for glassware cleaning.

4.4.3. Rinse the glassware thoroughly three times with reagent water. If the glassware is still visibly dirty or if spotting or beading occurs, repeat Section 4.4.2.

4.4.4. After air drying, the glassware should be spot- and stain-free. If not, then repeat the entire procedure.

4.4.5. Place the glassware in a designated clean storage location free from interferences or contamination.

4.5. Semivolatile Organics

4.5.1. If high-level contamination is suspected perform the appropriate high level cleaning before proceeding with the standard cleaning process described in Section 4.5.2 and following.

4.5.1.1. Non-polar organics: if the glassware was most recently *wet* with a solvent, then rinse three times with that solvent, if *wet* with water, then rinse three times with acetone. Collect rinses in the appropriate solvent waste container. Allow the remaining solvent to evaporate from the glassware in a ventilation hood to reduce analyst exposure to the solvent.

4.5.2. After using a piece of glassware, it is thoroughly rinsed with hot tap water and carried to the dirty glassware area. If it is visibly dirty, it should be washed or soaked immediately rather than allow the residue to harden and become more difficult to wash.

4.5.3. The glassware is immersed in a hot, soapy solution of water and laboratory detergent (recommended pH >10). An appropriate scrub brush is used to scrub the glassware. Change the wash water (when it becomes cold, visibly dirty or is used on glassware with oil or sediment residue) by emptying, rinsing the inside of the sink with hot water and refill with hot water and detergent.

4.5.4. Rinse the glassware three times vigorously with hot tap water. If the glassware is still visibly dirty, or if spotting or beading occurs, rinse with 1:1 HCl and repeat Section 4.5.3.

4.5.5. After completing the hot tap water rinse, rinse the glassware three times with deionized water and place it in a clean drying area.

4.5.6. Place cleaned glassware used for semivolatiles in a muffle oven for approximately one hour at 400°C. Do not heat glassware with visible residue. Baked-on residue is much more difficult to remove. Do not heat volumetric glassware, including flasks, pipettes, syringes, etc., to avoid deformation.

4.5.7. Clean glassware is inverted (where applicable) and stored prior to use in a designated clean storage location.

4.5.8. Before use, glassware is to be pre-rinsed with the solvent inherent to a given procedure if stored more than one week.

4.6. Volatile Organics

4.6.1. Rinse the glassware vigorously with hot tap water. If necessary, use a scrub brush to remove any remaining residue.

4.6.2. Place the cleaned glassware in the drying oven until dry or overnight.

4.6.3. Allow the cleaned and dry glassware to cool completely before use.

4.7. All Laboratory Groups

4.7.1. All glassware-washing brushes must be hung on hooks when not in use to prevent contamination and ensure proper drying.

5. DEFINITIONS

5.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current version.

6. POLLUTION PREVENTION

6.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution".

7. WASTE MANAGEMENT

7.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

7.2. Waste Streams Produced by the Method

7.2.1. The following waste streams are produced when this method is carried out.

7.2.1.1. Flammable Solvent Rinse. Used solvents generated from glassware cleaning operations are placed in waste containers identified as "Mixed Flammable Solvent Waste"

7.2.1.2. Spent Acid Rinse. Concentrated used acids generated from cleaning glassware are collected in containers identified as "Acid Waste".

8. REFERENCES

8.1. References

8.1.1. Corporate Quality Management Plan (CQMP), current version

8.1.2. TestAmerica Canton Quality Assurance Manual (QAM), current version

8.1.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

8.1.4. Revision History

Historical File:		Revision 1: 12/23/96		Revision 7: 05/06/08
		Revision 2: 10/25/99		Revision 8: 03/30/09
		Revision 3: 01/11/01		Revision 9: 04/28/10
		Revision 4: 06/22/01		
		Revision 5: 12/08/04		
		Revision 6: 02/16/07		

9. APPENDICES

9.1. See Table 1, Glassware Washing

Table 1 Glassware Washing

	Metals	Organics	Wet Chemistry
Wash	Hot water, detergent solution	Hot water, detergent solution	Hot water, detergent solution
Rinse	3 times tap water 1 time 1:1 Nitric acid 3 times reagent water	3 times tap water 1 time 1:1 Hydrochloric acid* 3 times reagent water	3 times reagent water
Dry	Air	Muffle at 400°C for one hour	Air
Storage	Designated cabinets and shelves	Designated cabinets and shelves	Designated cabinets and shelves

*If the glassware is visibly dirty after washing in a hot detergent solution, the glassware is rinsed with 1:1 HCl and rewashed with a hot detergent solution.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Canton

SOP No. NC-QA-018, Rev. 14
Effective Date: 12/10/13

Title: STATISTICAL EVALUATION OF DATA AND DEVELOPMENT OF CONTROL CHARTS

[Method: None]

Approvals (Signature/Date):

Rebecca Strait	11/12/13	Carolyne Kosack	10/31/13
Quality Assurance Manager	Date	Operations Manager	Date
	12/06/13		
Laboratory Director	Date		

This SOP was previously identified as SOP No. NC-QA-018, Rev 13, dated 05/31/12

Facility Distribution No. _____ **Distributed To: _____**

TABLE OF CONTENTS

1. PURPOSE

1.1. The purpose of this SOP is to describe the requirements for: 1) statistically establishing QC acceptance criteria, and 2) long-term trend analysis of QC data using control charts.

1.2. The control chart is an effective tool for long-term trending because it records in real time the accuracy (bias) and precision of the appropriate parts of the measurement process. The control chart provides the means to demonstrate statistical control.

1.3. This document accurately reflects current standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. RESPONSIBILITIES

2.1. Analyst

2.1.1. All QC data is entered into the Laboratory Information Management System (LIMS) for statistical evaluation for the generation of control charts. (Data entry may be automated.)

2.1.2. Monitor method performance using established limits and use the Control Chart Program in LIMS to identify and respond to any out-of-control situation. QC Data results are considered out of control when recoveries exceed established control limits.

2.2. Group Leader/Supervisor

2.2.1. Monitor method performance using established limits and use the Control Chart Program in LIMS to identify and respond to any out-of-control situation QA Department Staff.

2.2.2. For analytical methods, coordinate updating of control limits. During this process, review control charts to detect any trends in routine analytical procedures.

2.2.3. Archive control charts and statistically derived QC acceptance data.

2.2.4. Publish statistically derived QC acceptance criteria.

2.2.5. Provide guidance in the development of control charts and in the application of QC samples and acceptance data.

2.2.6. With the Laboratory Director and Operations Manager, ensure that Operations staff conforms to the requirements provided in this SOP.

2.3. Project Manager

2.3.1. Incorporate updated limits into project-specific QAPPs.

2.3.2. Submit project-specific control charts to the requesting client.

3. SAFETY

3.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

3.2. Normal office-dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in the laboratory area, appropriate personal protective equipment and precautions must be utilized.

3.3. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to a laboratory supervisor and the EH&S Coordinator.

4. PROCEDURE

4.1. Empirical Establishment of QC Acceptance Limits

4.1.1. The assessment of QC sample data shall be performed by comparing precision and accuracy results against control limits. As defined in the following subsections, the control limits used for this comparison shall be either in-house (statistically generated using historical data) control limits or published limits from methods, contracts, or project QA plans.

4.1.2. In-house limits for all QC data must be determined and compared to those limits published in the methods for applicable matrices. Some test methods specify interim control limits to be used until sufficient data points are available to generate in-house limits. These limits will be employed until in-house limits can be established. A minimum of 20 of the most recent data points should be used to establish in-house limits based on historical performance data for each major method. Periodically, QC data may need to be reviewed and house limits reestablished whenever a significant change in an analytical process occurs.

4.1.3. Control limits shall be generated for each matrix (i.e., aqueous, soil, and other matrices) for preparative methods, using data from at least 20 of the most recent data points. Limits are generated using a minimum three month timeframe to ensure the minimum numbers of points are included. A longer time period may be used to ensure the minimum numbers of points are included.

4.1.4. In-house control limits shall be established for the following samples:

4.1.4.1. Laboratory control sample (LCS) spike recoveries

4.1.4.2. Matrix Spike and Matrix Spike Duplicate (MS/MSD) spike recoveries

4.1.4.3. Surrogate spike recoveries in Method Blank and LCS for DoD samples

4.1.4.4. Surrogate spike recoveries in All Samples for routine work.

4.1.5. Control limits shall be established for all methods unless specified in project plans or regulatory methods.

4.1.6. The calculations used to generate the control limits for accuracy (%R) are described in the following subsections.

4.1.6.1. The %R is defined as the observed concentration in LCS divided by the theoretical concentration of the spike or LCS, times 100:

$$\%R = \frac{Found}{True} \times 100$$

4.1.6.2. The mean percent recovery and standard deviation is calculated using the following formulas:

$$\overline{\%R} = \frac{\sum_{i=1}^{n} \%R_i}{n}$$

$$S = \sqrt{\frac{\sum_{i=1}^{n} \left(\%R_i - \overline{\%R}\right)^2}{n-1}}$$

where,

%R	=	the mean percent recovery
$\%R_i$	=	the percent recovery of an LCS
n	=	the number of data points
S	=	the standard deviation of the data set of percent recoveries

4.1.6.3. The warning (95% or 2-sigma and control limits (99% or 3-sigma) are then calculated from the following equations:

Upper Control Limit = $\overline{\%R}$ + 3 s

Lower Control Limit = $\overline{\%R}$ - 3 s

Where,

$\overline{\%R}$ = the mean percent recovery
s = the standard deviation of the data set

4.1.7. Control limits will be recalculated after excluding the following points from the calculations:

4.1.7.1. Samples with values outside control limits due to assignable cause.

4.1.8. The LIMS is equipped to perform a Grubbs outlier test as an optional tool for evaluation.

4.2. Control Chart Generation

4.2.1. A control chart (X chart) is generated to monitor accuracy and precision by plotting the LCS %R data in a graphical format as follows.

4.2.1.1. The average of the %R determinations for the original data set is established as the midpoint on the Y axis of the graph.

4.2.1.2. The upper and lower warning and control limits are plotted as solid horizontal lines across the graph at their respective points on the Y axis.

4.2.1.3. The calculated %R of each spiked sample is plotted chronologically on the graph to determine whether the recovery is within the warning and control limits of the control chart.

4.2.2. Control charts can be generated from LIMS Control Chart module.

4.2.3. The following information must be present on the control charts or in an associated table.

4.2.3.1. Parameter, analytical method, and preparation procedure

4.2.3.2. LCS Batch ID allowing cross-referenceto LIMS containingall analytical information

4.2.3.3. Matrix

4.2.3.4. Number of points used

4.2.3.5. Mean

4.2.3.6. Standard deviation

4.2.3.7. Percent recoveries

4.2.3.8. Upper and lower control limits

4.2.3.9. Chart generation date

4.3. Evaluation of Control Charts

4.3.1. Criteria for an Out-of-ControlCondition

The causes for a shift or a trend in control charts could result from many reasons, including, but not limited to:

- incorrect preparation of a standard or a reagent
- sample contamination
- improper storage or preservation
- incorrect instrument calibration
- poor analytical technique
- deviation from the analytical method

4.3.1.1. These conditions may indicate that the measurement system is out of statistical control. When this situation occurs, the data must be evaluated thoroughly to identify the most appropriate corrective action to be implemented. The problem and its solution may be documented as appropriate. In reviewing control charts, any significant changes in key analysts, instrumentation, standard reference materials, or processes must be kept in mind to explain potential out-of-control situations. After thorough evaluation of the data and documentation of corrective actions taken, the QA Department must determine if the defensiveness of analytical results generated during the out of control situation has been jeopardized. If it is determined that data defensivenesshas been compromised, the client will be notified of the out-of-control situation. Limits that are outside method limits need further investigation since it may indicate a system is out of control.

4.3.1.2. Recoveries greater than 150 % and less than 10% will be excluded from the limit calculation.

4.3.1.3. When reviewing MS/MSD and Surrogate charts, all dilutions greater than 4X will be excluded from the limit calculation.

4.3.2. Laboratory Review of Control Charts and Control Limits

4.3.2.1 After QA evaluation, the new control limit charts are forwarded to the laboratory staff for review. Limits will be updated in LIMS within one week of generation. Update in LIMS

4.3.3. Control limits are updated in LIMS after approval. Limits are active on the day of approval in LIMS.

4.4. Setting Control Limits

4.4.1. The working control limits to be used by the laboratory are based on evaluation of the calculated laboratory statistical performance and available inter-laboratory limits provided in the reference methods. Note that some SW-846 methods only supply single-operator or single-laboratory method performance data, which may not be appropriate. Recommended minimum and maximum limits are listed below. Note that some analytes may exhibit recovery levels outside of these recommendations. Analyst discretion may be used in setting recovery limits for poor performing analytes.

4.5. Recommended Min/Max Limits

QC Limit	Minimum	Maximum
Lower Control Limit (LCL)	10% MS/MSD 40% LCS/Surrogate	80%
Upper Control Limit (UCL)	120%	160%
Relative Percent Difference (RPD)	10%	35% water 40% solid

5. DEFINITIONS

5.1 Control Chart: A graphical QC tool to monitor method performance over time and to establish acceptance limits.

5.2 Relative Percent Difference (RPD): A measure of intra-lab precision based on a

duplicate sample analyses.

5.3 Grubbs Test: Extension of sample sizes and percentage points for significant tests of outlying observations - a statistical outlier test.

5.4 Percent Recovery (%R) or Recovery: A measure of the accuracy (bias) of the measurement process based on a comparison of a measured value for a fortified (spiked) QC sample against the known spiked values.

5.5 Precision: A measure of mutual agreement (or variability) among individual measurements of the same property, usually under prescribed similar conditions.

5.6 Accuracy: The degree of agreement of a measurement (or an average of measurements of the same thing) with an accepted reference or true value. Accuracy is the measure of bias inherent in the system.

5.7 Bias: A systematic (consistent) error in test results. The difference between the population mean and the true or reference value, or as estimated from sample statistics; the difference between the sample average and the reference value.

5.8 X-chart: A control chart that plots a single measurement of a property (e.g., percent recovery) of quality control samples over time. The chart consists of a single line that is the mean of the statistic, warning limits at ± two standard deviations, and control limits at ± 3 sigma.

5.9 Assignable Cause: A known reason for an outlying result (e.g., no spike added).

5.10 Duplicate: A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

5.11 Laboratory Control Sample (LCS)

5.11.1 Organics: An LCS is a volume of deionized laboratory water (for water samples) or a suitable solid material (e.g., clean sand) (for soil/sediment/othermatrix samples) which is spiked with compounds of interest and subjected to the entire analytical procedure in order to estimate the accuracy of the method via percent spike recovery.

5.11.2 Inorganics: A well-characterized liquid or solid sample which is prepared, digested or extracted along with each analytical batch of samples.

6 REFERENCES

6.1 References

6.1.1 TestAmerica Canton Quality Assurance Manual (QAM), current version

6.1.2 Test Methods for Evaluating Solid Waste, Third Edition, SW-846, US EPA, Final Update III, December 1996

6.1.3 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

6.1.4 Corporate Quality Management Plan (CQMP), current version

6.1.5 Revision History

Historical File:		Revision 4: 06/19/00		Revision 11: 03/14/11
		Revision 5: 05/03/01		Revision 12: 05/31/11
		Revision 6: 07/30/01		Revision 13: 05/31/12
		Revision 7: 12/08/04		
		Revision 8: 02/15/07		
		Revision 9: 05/20/08		
		Revision 10: 02/04/10		

6.2 Associated SOPs and Policies, current version

6.2.1 QA Policy, QA-003

6.2.2 Supplemental Practices for DoD Project Work, NC-QA-016

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Canton

SOP No. NC-QA-021, Rev. 10
Effective Date: 9/30/13

Title: EVALUATION OF METHOD DETECTION LIMITS FOR CHEMICAL TESTS

[Method: None]

Approvals (Signature/Date):

Operations Manager — 09/17/13 Date

Quality Assurance Manager — 09/30/13 Date

Laboratory Director — 09/21/13 Date

This SOP was previously identified as SOP No. NC-QA-021, Rev 9, dated 04/30/12

Facility Distribution No. _____ **Distributed To: _____**

TABLE OF CONTENTS

1. PURPOSE

1.1. The document describes the policies and procedures relative to the evaluation, acceptance and iterative process of method detection limits for chemical tests. This SOP is based on TestAmerica Policy CA-Q-S-006 (Method Detection Limit Studies) and 40 CFR Part 136 (Appendix B).

1.2. This document accurately reflects current Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. RESPONSIBILITIES

2.1. It is the responsibility of the employee to perform the procedure described herein in full compliance with this SOP.

2.2. It is the responsibility of the Laboratory Director, QA Manager, and departmental Supervisor of this facility to assure that the policy and analyses described are performed in full compliance with this SOP and the corresponding analytical SOPs. It is also their responsibility to supply adequate training, materials, and equipment to enable the employee to perform this SOP correctly.

3. SAFETY

3.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the facility Addendum to the Corporate EH&S Manual, and this document.

4. PROCEDURES

4.1. Any deviations from this procedure must be documented as a nonconformance with a cause and corrective action described.

4.2. Method Detection Limit Study (MDL Study) Evaluation

4.2.1. MDL samples are logged into LIMS. Each of the replicate spikes is extracted or digested in the same manner as samples. Routine cleanup steps are included.

4.2.2. MDL analytes must be detectable by the instrument and meet all qualitative criteria specified by the method or SOP.

4.2.3. There must be a minimum of 7 replicates. All replicates must be reported, unless a known physical reason is noted. To reject an MDL point, it must be determined that there is a valid reason for rejection the point, as would be

applied to sample results. Rejecting points must be supported by sound, documented and technically based justification.

4.2.4. Results are completed using the Control Chart function in LIMS. MDLs are representative of the performance of the method in the laboratory and can be influenced by the performance of individual instruments.

4.2.5. If more than 7 replicates are used for MDL Studies, data from all replicates must be used in calculating the MDL. In these cases, the appropriate student's t-value (n-1 degrees of freedom, 99% confidence level) must be used in the calculation of the MDL (see 40 CFR Part 136, Appendix B). MDLs may be determined on a matrix, method, and instrument-specific basis.

4.2.6. Refer to SOP CA-Q-S-006 for details concerning the frequency of analysis.

4.3. MDL Study Acceptance Criteria

4.3.1. See 40 CFR Part 136, Appendix B for MDL acceptance criteria.

4.3.2. Detection Limit studies may be evaluated against any associated IDL evaluations, previous MDL studies, established RLs, any method specified IDLs or MDLs, and any other criteria in TestAmerica North Canton Quality Assurance Manual (QAM), Supplemental Practices for DoD Project Work SOP (NC-QA-016), and MDL Policy (CA-Q-S-006).

4.3.3. All studies must be reviewed by appropriate QA personnel and laboratory management staff prior to data release.

5. DEFINITIONS

5.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current version

6. DATA ANALYSIS AND CALCULATIONS

6.1. Data review

6.1.1. Technical review

6.1.1.1. MDL studies are logged into LIMS. The laboratory prepares and analyzes the study and enters the result into LIMS.

6.1.1.2. The QA Department is notified via LIMS when final studies are complete.

6.1.1.3. The MDL results are calculated using the Control Chart function in LIMS. Refer to Work Instruction WI-NC-96A for direction on using the program. Ratios must be between 1 and 10 and MDLs must be less than the standard reporting limits. If any spike-to-MDL ratio exceeds 10, corrective action may be required, including repeating the MDL study for the affected analytes.

6.1.1.4. All MDL results must be verified prior to use by an MDL Verification standard. These are known as LOD samples for DoD work and may be performed concurrently with or immediately after each MDL study.

6.1.1.5. LODs are prepared at approximately 2-4 times the MDL concentration in the same media as was used for the original MDL study, and are subject to the entire preparation and analysis process. For methods with a large number of analytes and a wide range of MDL concentrations, this will require processing LODs at multiple concentrations. For example, LODs for Method 8270 must often be prepared at as many as five concentrations.

6.1.1.6. LODs are performed on every instrument that will be used for DoD work on a quarterly basis.

6.1.1.7. Peaks for the LOD standards must be integrated in the same manner as are samples. It is not acceptable to finesse the LOD to lower levels using special manual integrations.

6.1.1.8. If an LOD standard is not detected at the first concentration, repeat the verification at approximately twice the previous concentration. The doubling process is repeated as necessary. The final MDL is established at the concentration of the lowest passing MDLV. The new LOD is established at 2-4 times this value.

6.1.1.9. Additional Requirements for MDLs are listed in SOPs NC-QA-016 and CA-Q-S-006.

6.2. Calculations

Equation 1. Standard deviation (SD).

$$SD = \sqrt{\sum_{n=1}^{n} \frac{(RF_n - \overline{RF})^2}{n-1}}$$

Equation 2. MDL analyte recovery.

$$\%Recovery = \left(\frac{Amount\ found}{Amount\ spiked}\right) \times 100$$

Equation 3. % relative standard deviation (%RSD).

$$\%RSD = \frac{Standard\ Deviation}{\overline{RF_i}} \times 100$$

Equation 4. Calculation of MDL.

$$MDL = Standard\ deviation\ x\ t_{(n-1,\ 0.99)}$$

Where:

$t_{(n-1,\ 0.99)}$ is the one-sided Student's t value appropriate for a 99% confidence level with n-1 degrees of freedom. 40CFR part 136, Appendix B provides critical values of t for the number of replicates supported by this application.

Equation 5. Calculation of spike-to-MDL ratio. Note that both values must have the same units.

$$Spike\text{-}to\text{-}MDL\ ratio = \frac{Spike\ amount}{MDL}$$

6.3. Detection Limits Adjusted for Long-Term Blanks

6.3.1. For any tests that frequently produce positive blank results, it is advisable to determine if detection limits need to be adjusted for long-term blanks. For metals analyses, it is required in order to meet the DoD ICB and CCB requirements. In these situations, the final verified MDL used for reporting purposes should be greater than long-term blank concentration. If not, the MDL is elevated to the 99% upper confidence limit of long-term blank results.

$$99\%UCL = m + t * s$$

where:

m = mean concentration for uncensored blank results

s = standard deviation

$t_{(n-1,\ \text{one-tail},\ 1\ \alpha=0.95)}$ is the students t value appropriate for a 95% upper confidence level and n-1 degrees of freedom

7. REFERENCES

7.1. References

7.1.1. 40CFR part 136, Appendix B

7.1.2. TestAmerica Canton Quality Assurance Manual (QAM), current version

7.1.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

7.1.4. Corporate Quality Management Plan (CQMP), current version

7.1.5. Revision History

Historical File:		Revision 3: 10/04/00		Revision 8: 03/15/11
		Revision 4: 11/28/04		Revision 9: 04/30/12
		Revision 5: 04/25/07		
		Revision 6: 05/15/08		
		Revision 7: 02/15/10		

7.2 Associated SOPs and Policies, current version

7.2.1 Method Detection Limit Studies, and CA-Q-S-006

7.2.2 Supplemental Practices for DoD Project Work, NC-QA-016

7.2.3 TALS Control Chart for MDL, MDLV and LOQ Reports, WI-NC-96A

Student's t, Single-tail, 95% Upper Confidence Limit Table

Number of Replicates	Degrees of freedom (n-1)	Students t value
21	20	1.725
31	30	1.697
41	40	1.684
51	50	1.676
61	60	1.671
71	70	1.667
81	80	1.664
91	90	1.662
101	100	1.660
121	120	1.658
8	8	1.645

DoD Detection Limit Example

A single-day aluminum MDL study was performed on each of four ICP instruments:

Instrument # 16 ➔ 10.9 ug/L

Instrument# 20 ➔ 6.5 ug/L

Instrument# 21 ➔ 4.4 ug/L

Instrument# 22 ➔ 0.71 ug/L

The highest aluminum MDL for all instruments = 10.9 ug/L.

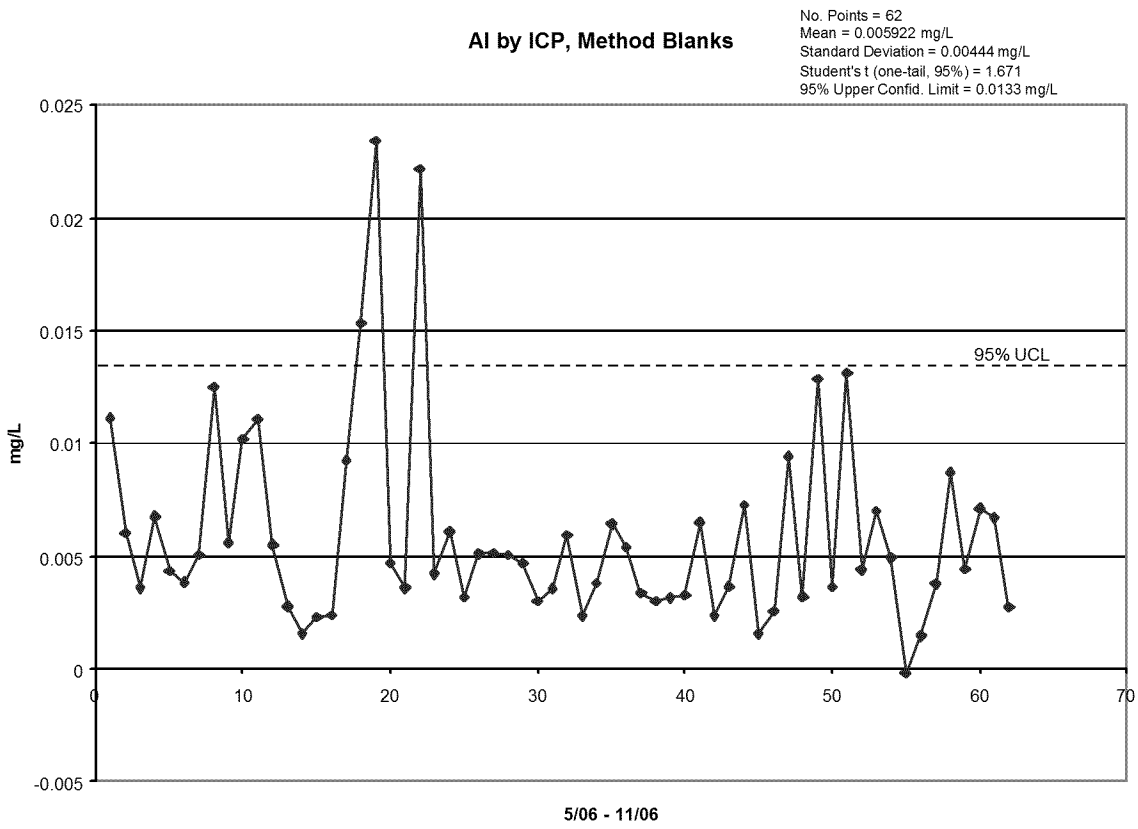
The 95% upper confidence limit was calculated using six months of aluminum data (see chart that follows):

95% UCL = 13 ug/L

The long-term blank is higher than the worst-case MDL. Therefore,

DoD MDL = 13 ug/L

LONG-TERM BLANK DATA EXAMPLE



TestAmerica
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TestAmerica Canton

SOP No. NC-QA-029, Rev. 4
Effective Date: 9/30/13

Title: NONCONFORMANCE AND CORRECTIVE ACTION SYSTEM

[Method: None]

Approvals (Signature/Date):

Caralyne Raach 09/18/13
Operations Manager Date

Rebecca Strait 09/30/13
Quality Assurance Manager Date

09/21/13
Laboratory Director Date

This SOP was previously identified as SOP No. NC-QA-029, Rev 3, dated 04/23/12

Facility Distribution No. _____ **Distributed To: _____**

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1. PURPOSE

1.1. The purpose of this document is to establish procedures for the identification and documentation of nonconformances and the corrective actions taken as a result of these events. The TestAmerica Canton Quality Assurance Manual (QAM) requires documentation of instances of deviations from established control limits, approved standard operating procedure (SOPs), or client-specified requirements. The Nonconformance Memo (NCM) described in this procedure is used to document deviations from TestAmerica policies and procedures and documented client specifications including root causes and corrective actions taken to remedy the nonconformance. The NCM is stored in electronic form in LIMS.

1.2. This document applies to procedures, services, analytical data, reports, or materials purchased by the laboratory or supplied by the laboratory to its clients.

1.2.1. Sample receiving exceptions:

1.2.1.1. Some nonconformance anomalies related to sample receiving activities, such as sample ID, temperature and preservation discrepancies, may be documented separately from the NCM process by use of the Cooler Receipt Form (CRF) as described in a facility-specific SOP.

1.2.1.2. HTVs and other observed deficiencies that impact data quality or reporting must be documented in LIMs by sample receiving personnel.

1.2.2. Regardless of the type of system used to handle the sample receiving and client-related issues, including holding time violations (HTVs), the system must include and emphasize the immediate notification of the Project Manager (PM). This will allow the PM to initiate immediate client notification and resolution of how to proceed. See Section 5, Definitions, for further clarification of application.

1.3. Nonconformances can be identified by TestAmerica laboratory employees in the course of their daily operations or by external parties (i.e., customers and representatives of customers) through review of records, audit, or proficiency testing.

2. RESPONSIBILITIES

2.1. **Laboratory Analyst/Technician:** During the course of their work, all employees are responsible for creating a Nonconformance Memo to identify and document problems that might affect the quality of TestAmerica's product. Analysts should also identify or attempt to seek out possible measures to correct the problem. By signing or initialing laboratory notebooks, forms, bench sheets, data reports, and other quality-related documents, employees are verifying that procedures have been followed. Any deviation that might render a measurement suspect must be documented.

2.2. **Group Leader:** Each Group Leader is responsible for the review of NCMs to ensure that problems which might affect data quality are adequately described and that personnel are assigned to correct them. Together with Project Managers and Quality Assurance personnel, Group Leaders are responsible for determining the appropriateness of planned corrective actions.

2.3. **Project Manager (PM):** The Project Manager is responsible for relaying project requirements to staff so that special project requirements are understood and nonconformances recognized. The Project Manager communicates conformance problems to clients and documents decisions made with clients. The Project Manager ensures that short-term corrective actions for routine analytical QC failures are completed. An example would be making sure that repreparation and analysis of a sample was done. The Project Manager can and must withhold final reports to clients until corrective actions agreed to with the client have been completed.

2.4. **QA Manager:** The Quality Assurance Manager or designee is responsible for periodically reviewing NCMs to ensure that actions taken are appropriate, and assisting in resolving QA/QC discrepancies. The NCM system in TALS can also be used to monitor for trends that might indicate long-term quality problems. Systematic problems are investigated, NCMs issued and reviewed, and spot audits conducted to ensure that long-term corrective actions have been successfully completed. If review of an area reveals a significant problem with data quality, the Quality Assurance Manager has the authority and responsibility to stop production in that laboratory area.

2.5. **Operations Manager/Technical Director**: The Operations Manager and/or Technical Director shall ensure corrective actions are technically appropriate and have been implemented. Along with the Laboratory Director and Quality Assurance Manager, the Operations Manager and Technical Director shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work.

2.6. **Laboratory Director:** The Laboratory Director shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work. The Laboratory Director is also responsible for the implementation of the NCM system in the laboratory.

3. SAFETY

3.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

3.2. Normal office-dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.

3.3. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to a Laboratory Group Leader and the EH&S Coordinator.

4. PROCEDURE

4.1. When to Initiate a Nonconformance Memo

4.1.1. Lab associates are to initiate an electronic nonconformance memo (NCM) whenever procedures, data, or standard materials deviate from established specifications. All nonconformances require an NCM (see definitions of nonconformance, anomalies, and deficiencies in Section 5 and Section 1.2 for exceptions).

4.1.2. All Standard operating procedures (SOPs) shall be followed. By signing or initialing laboratory notebooks, bench sheets, data reports, and other quality-related documents, employees are verifying that the SOPs have been followed with the exceptions of the pre-approved deviations (as described in QAPPs or equivalent systems). Any intentional deviation from an SOP must be pre-approved by the Analytical Group Leader and must be documented using the NCM process. Questions about the acceptability of a deviation can be forwarded to a member of the Quality Assurance staff for review.

4.1.3. An NCM is to be completed for each instance of a nonconformance. A single NCM can be used for a single event affecting multiple job numbers and samples, but normally a separate NCM would be initiated for different nonconformance issues.

4.2. The analyst creates an NCM using the NCM Module in TALS

4.2.1. Group Leader Review and Approval

4.2.1.1. The Group Leader receives notification of the NCM via e-mail. The Group Leader reviews the NCM in TALS.

4.2.2. Project Manager Review, Client Notification, and Project Documentation

4.2.2.1. The Project Manager will determine if client notification is required to get client input on the appropriate corrective action or to notify the client of problems related to sample analysis.

4.2.3. Quality Assurance Review and Trending

4.2.3.1. Designated QA staff shall review NCMs periodically for conformance with standard laboratory practices.

4.2.3.2. The QA staff will review the NCM Management Report in TALS to identify repetitive quality issues that may be systematic in nature and may require root cause analysis and corrective actions to prevent recurrence. Recurrent technical or Information Technology problems shall be referred to the appropriate technical group for root cause analysis and corrective actions. Correction of systematic problems could take the form of modifications of nonconforming procedures, repair or replacement of deficient equipment, training or replacement of personnel. Findings, root causes and corrective actions from these investigations or audits shall also be documented in a corrective action log maintained by the QA department. Resolution of corrective actions for systematic problems, along with supporting evidence, must be documented by the responsible laboratory area. The QA staff will conduct follow-up assessments to confirm that correction of systematic problems is successful.

4.2.4. Instrument/Equipment Tag Out

4.2.4.1. Instruments and equipment, which habitually fail to meet calibration criteria or are out of service due to needed repair or other reasons, must be marked with a clearly visible tag or sign indicating the nonconforming condition (see example in Attachment A) A Tag Out sign is needed if the repair exceeds an 8 hour shift. Equipment that is no longer in use must be tagged out and removed from the laboratory.

4.2.4.2. The corrective action will be to either permanently remove the instrument from service or to have the instrument repaired. If an instrument is repaired, its reliability must be demonstrated through successful recalibration before the nonconformance can be closed. The tag remains in effect during the demonstration period.

4.2.4.3. Records of all maintenance activities, including return to control details and date, must be documented in the instrument maintenance logbook.

5. DEFINITIONS

5.1. ***Nonconformance:*** An unplanned deviation from an established protocol or plan. The deviation may be the result of TestAmerica actions--then termed a **deficiency**--or the result of events beyond the control of TestAmerica--then termed an **anomaly**.

A nonconformance exists when:

5.1.1. Any laboratory QC sample (e.g., method blank, laboratory control sample, duplicate laboratory control sample, matrix spike, matrix spike duplicate, and surrogate spike) component in which the result is outside established control

limits and demonstrates a **systematic** deficiency. Any matrix spike or matrix spike duplicate or sample related QC outside of established control limits attributed to matrix effects must be documented.

5.1.2. A procedure is not performed as described in the applicable SOP or QA Policy, ***except*** in cases where the procedure has been performed according to a client-specified document TestAmerica has agreed to follow (e.g., EPA SOWs and QAPPs).

5.1.3. A practice or procedure is not performed as described according to a client or project document that TestAmerica has agreed to follow.

5.1.4. Purchased materials or services are determined to be defective and their use would affect data quality.

5.1.5. Holding time violations occur regardless of what or whose actions caused them.

5.1.6. A formal NCM is not required for routine instrument maintenance and malfunctions, which can be documented in instrument maintenance logbooks.

5.2. ***Root Cause:*** The most fundamental reason for the failure or inefficiency of a process.

5.3. ***Root Cause Analysis:*** A class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variations in performance or the occurrence of a significant failure.

5.4. ***Corrective Action:*** Measures taken to rectify conditions adverse to quality and, where possible, to prevent their re-occurrence.

5.4.1. Corrective actions may vary from reporting the data as is with appropriate documentation to a complete re-evaluation and restructure of a system.

5.4.2. Many corrective actions can be implemented immediately; however, some will take time to implement.

6. MISCELLANEOUS

6.1. Associated Reference Documents

6.1.1. TestAmerica Canton Quality Assurance Manual (QAM), current version

6.1.2. Corporate Quality Management Plan (CQMP), current version

6.1.3. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version

6.1.4. Revision History

Historical File:		Revision 0: 12/11/95		
(formerly CORP-QA-0010)		Revision 1: 08/24/97		
		Revision 2: 06/15/99		
		Revision 3: 09/19/07		Revision 3: 04/23/12
		Revision 0: 09/24/08 (NC-QA-029)		
		Revision 1: 11/10/09		
		Revision 2: 04/12/11		

6.2. Appendices

6.2.1. Attachment A: Instrument/Equipment Nonconformance Tag Form

ATTACHMENTA

EXAMPLE

INSTRUMENT/EQUIPMENT NONCONFORMANCE TAG OUT FORM

TESTAMERICA

CAUTION

DO NOT USE

NONCONFORMING ITEM

AFFECTEDITEM _____

_____

_____

_____ _____
ANALYST DATE

WORK MAY NOT PROCEED ON THIS ITEM UNTIL
SUCCESSFUL CALIBRATION IS DOCUMENTED.

TestAmerica
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North Canton

SOP No. NC-SC-005, Rev. 6.9
Effective Date: 3/27/12

Title: SAMPLE RECEIVING

[Method: None]

Approvals (Signature/Date):

Technology Specialist	03/21/12 Date	Health & Safety Coordinator	01/13/12 Date
Quality Assurance Manager	02/6/12 Date	Laboratory Director	01/12/12 Date

This SOP was previously identified as SOP No. NC-SC-005, Rev 6.8, dated 08/18/10

Facility Distribution No. _____ **Distributed To: _____**

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1. SCOPE AND APPLICATION

1.1. It is the responsibility of the Sample Receiving Department personnel to perform the procedures described herein in full compliance with this SOP.

1.2. It is the responsibility of the Laboratory Director, QA Manager, Operations Manager, and Departmental Supervisor of the facility to assure that the procedures described are performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable personnel to perform this SOP correctly.

1.3. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. Not applicable

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Not applicable.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn at all times when at the counter while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves are worn for tasks that present a strong possibility of getting cut. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment (PPE) and precautions must be utilized. Disposable gloves that have been contaminated will be removed and discarded.

5.3. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure/Unusual Hazards
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Hydroxide	Corrosive	2 mg/m^3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Exposure to chemicals must be maintained as low as reasonably achievable. If samples are known to be hazardous, they are opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation, where possible. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Thermometers

6.2. PPE such as gloves, lab coats, safety glasses, etc.

6.3. Utility knives

6.4. pH paper

6.5. Copier, printer, computer and label generator

6.6. Carts

7. REAGENTS AND STANDARDS

7.1. Not applicable

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Not applicable

9. QUALITY CONTROL

9.1. Nonconformance and Corrective Action

9.1.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Not applicable

11. PROCEDURE

11.1. Receiving and Unpacking Samples

11.1.1. Any deviations from this procedure must be documented as a nonconformance with a cause and corrective action described.

11.1.2. The procedures listed in this document describe the responsibilities of Sample Receiving personnel in ensuring that data is transmitted correctly from the client to all personnel involved with sample analysis and review.

11.1.3. Samples typically arrive packed in coolers or foam boxes. Upon receipt, the coolers are triaged based on rush status (TAT: turn-around time) and expirable tests listed on the Chain-of-Custody (COC). Refer to Appendix I for a listing of expirable tests.

11.1.4. The following flagging system is used to show the priority level associated with triaged samples:

- 24 hour TAT – flag with red folder
- 48 hour TAT – flag with blue folder
- 72 hour TAT – flag with yellow folder
- 1 week TAT – flag with green folder
- Encore or low-level mercury samples – flag with orange paper with "Encore", "LLHg", or MeHg written on it

11.1.5. If the COC lists expirable tests, the cooler is marked with a manila folder.

11.1.6. Rush and expirable coolers are to be unpacked first and logged as soon as possible. The lab groups are to be notified with any rush TAT requiring 72 hours or less. Samples requiring a standard TAT and/or no expirable tests are given lowest priority.

11.1.7. The following information is documented on the Cooler Receipt Form (CRF). Refer to Appendix II for a current version.

11.1.7.1. Client name

11.1.7.2. Project name or number (if known)

11.1.7.3. Signature of Sample Receiving Technician

11.1.7.4. Date cooler received and opened

11.1.7.5. Method samples were received (overnight courier, client drop-off, or other means)

11.1.7.6. Cooler ID number (if known)

11.1.7.7. Type of container received (TestAmerica cooler, client cooler, foam box)

11.1.7.8. Presence of the custody seals on the outside of the cooler

11.1.7.9. Presence of custody seals on bottles

11.1.7.10. Presence of the custody papers (i.e., COC)

11.1.7.11. Verification that custody papers were properly filled out (ink, signed, match labels)

11.1.7.12. Verification that custody papers were relinquished by the client

11.1.7.13. Presence of a packing slip

11.1.7.14. Presence and type of packing material information

11.1.7.15. Cooler temperature upon receipt. The temperature of the cooler is taken by an IR gun. The IR gun is directed at the label (minimizes laser reflection) of a sample bottle or temperature vial, whichever best reflects the cooler temperature. According to federal regulations, the temperature upon receipt should be $\leq 6°C$. The Project Manager (PM) is contacted (and the anomaly is narrated) when the temperature is $> 6°C$ or if samples are frozen.

11.1.7.16. Condition of bottles upon receipt (good condition, broken, etc.)

11.1.7.17. Complete bottle labels (date, time, client ID)

11.1.7.18. Information on bottle labels and tags agree with custody papers

11.1.7.19. Verification that the correct bottles were received for the tests indicated (refer to Appendix III)

11.1.7.20. Volatile (VOA) vials were checked for the presence of air bubbles. If vials have bubbles exceeding 6 mm in diameter, it is narrated and the Project Manager (PM) is contacted.

11.1.7.21. Verification that sufficient amount of sample was received in order to perform the tests listed on the COC.

11.1.7.22. Verification of pH for preserved samples (except Volatiles and TOC) upon receipt. An aliquot of sample is removed from the sample container by using a disposable glass Pasteur pipette, and placing a drop of the sample onto a pH paper strip. The pHs are then recorded on the CRF. The pH paper strips are then discarded.

11.1.7.22.1. Purchased prepared vials of preservatives (refer to Appendix IV) are used if samples are not at the correct pH. The pH is adjusted by adding the appropriate preservative in 5 mL increments up to a maximum of 20 mL per liter of sample or unless there is a reaction. The pH adjustment and final pH are noted on the CRF. The Lot Number of the pre-made preservative is noted on the CRF. It is the responsibility of the Sample Receiving group to change the lot number when a new shipment arrives and to document when a new box is opened. Receiving will adjust the pH under a hood to prevent hazards to employees in case a sample off-gases noxious fumes.

Note: For metals samples that require preservation, the time of acid addition must be noted. The sample must be held for 24 hours prior to analysis. The concentrations of the preservatives used are as follows:

4N Sodium Hydroxide
1N Zn Acetate
1:1 HCL (18%)
1:4 $HN0_3$ (18%)
1:2 H_2SO_4 (33%)

11.1.7.23. The presence or absence of a Trip Blank and if VOA samples were received.

11.1.7.24. Documentation (name of PM, date, name of Sample Receiving technician, method used to contact PM) that the PM was notified of any anomalies associated with receipt of a project.

11.1.8. The Sample Receiving technician removes all sample containers. Any broken, leaking, or dirty sample containers are to be placed inside the fume hood. Dirty sample containers are to be cleaned appropriately, so as not to contaminate the sample storage area. Completely broken or unsalvageable samples are disposed of properly.

11.1.9. Coolers emitting strong vapors/fumes when opened will be taken to the Metals Lab and unpacked in either of its hoods.

11.1.9.1. Any problems concerning exposure while unpacking samples must be immediately reported to the Group Leader and/or the EH&S Coordinator.

11.1.10. Volatile sample(s) suspected (e.g., odor) or known (client information or site history) to be high in volatile concentration is stored in a separate designated volatile area.

11.1.11. The Sample Receiving technician examines all documents and compares information on the sample container labels to the COC. All discrepancies are documented on the CRF.

11.1.12. Problems or discrepancies that compromise sample integrity (such as limited sample volume, sample identification which cannot be determined from the COC, incorrect pH levels, or preservatives, if known, or broken leaking samples) are reported to the PM. The PM will advise Sample Receiving on how to proceed.

11.1.13. Expirable tests (hold time 48 hours or less) must be written on the top of the bottles from which they are to be analyzed. If more than one method exists for the analysis, the method must also be written.

11.1.13.1. Sample containers are placed in the red bin designated for expirables in the Sample Receiving room. The Label ID or sample ID must be recorded on the expirable logsheet along with the record of the test to be run, special method, if necessary, and the initials of the person relinquishing the sample. The Wet Chem Lab group checks this bin throughout the day, and is responsible for signing out the sample container when they take it. Sample Receiving will also turn on the "light" to alert Wet Chem there are expirables in the bin.

11.1.13.2. At the time of sample receipt, the sample must be inspected to determine the correct method reference for pH analysis. General Chemistry staff members will determine the percent of aqueous phase present and notify the Sample Receiving staff if a method change is needed. The Sample Receiving staff will notify the Project Manager to make the appropriate change in LIMS. Method 9040B should be used if the aqueous phase constitutes at least 20% of the total volume of the waste. Method 9045C should be used for measuring pH in soils and waste samples. If water is present, it must constitute less than 20% of the total volume of the sample.

11.1.14. Samples requiring a Total Solid (TS) result are split off for analysis. Splitting these samples is the responsibility of the Sample Receiving group. A small representative portion (approx. 5-10 grams) from each sample is placed into a small plastic snap-top container designated for the TS analysis. If the sample has two or more layers, an aliquot is taken from each layer. If the sample appears to be homogenous, an aliquot is taken from the center of the sample. If the TS container is not labeled with a LIMS label when the aliquot is removed, the TS container must be labeled with handwritten client ID. Containers designated for volatiles analyses (VOC) must not be opened in Sample Receiving. If only one container is received and volatiles are requested, the Receiving group must **not** split off because of possible contamination or possible lost volatiles. An empty TS container with a LIMS label is given to the VOC Analysis group for each solid. The VOC group will aliquot for the TS when they open the container for analysis. The TS plastic containers are placed in baggies according to lot number, and are stored in bins inside the walk-in cooler door.

11.1.15. When samples need to be composited, the following procedure is followed, unless there are specific instructions from the client.

11.1.15.1.1. Equal aliquots are weighed from each container and mixed thoroughly and transferred to a new container.

11.1.15.1.2. The amount aliquoted (in grams) is recorded on the Cooler Receipt Form.

11.1.16. The Sample Receiving technician who unpacked the cooler must sign the COC in the appropriate place.

11.1.17. Samples received after hours are signed for by a TestAmerica North Canton employee and placed in the walk-in cooler to be processed the following business morning.

11.2. Sample Log-In

11.2.1. Samples are logged in LIMS after unpacking has been completed, and all anomalies have been documented.

11.2.2. All COCs are photocopied, and given to the associated PM; who in turn, provides the appropriate project numbers.

11.2.3. A project number must reflect what is on the Chain-of-Custody. Any discrepancies must be resolved by the PM. Projects are generally not logged until the PM provides a project number. However, if it is a routine project and the project number is known, log-in can proceed.

11.2.4. In the event a project cannot be logged due to discrepancies, all associated paperwork is placed into a black folder. All samples and the black folder are put into cold storage. These projects are recorded on the dry-erase board posted on the walk-in cooler door.

11.2.5. Log-in is initiated by retrieving the paperwork (including a photocopy of the COC or an email from the PM, and project number) from the unpacked projects on the counters. The following information is included at log-in cooler temp, cooler ID, packaging, cooler type, client sample ID, sample collection date and time, sample matrix (example: WW = wastewater, WG = groundwater, SO = solid), sample type (example: Normal = N1, Trip Blank = TB, Rinse Blank = RB1), and the anlyses requested on the COC.

11.2.5.1. A unique job number is generated for each project that is logged. The job number is sequential and increases by a single number every time a new job is started.

11.2.5.2. North Canton jobs are designated by the numbers 240-xxxxxx, Pittsburgh is designated by the numbers 180-xxxxxxxx etc..

11.2.6. If a client requests an MS/MSD for a sample(s), it is denoted in LIMS by an "MS" for the MS and a "MSD" for the MSD.

11.2.7. Each container is given an individual letter followed by the sample number (Example: "1001-A-1, 1001-A-2", etc.).

11.2.8. A Level One review of the log-in is to be performed by the Sample Control technician in charge of logging in the project. A Level Two review is to be performed by a different Sample Control technician. **Level review is mandatory before the summary is delivered to the Project Manager.**

11.3. Labeling

11.3.1. After log-in is completed, the sample containers are labeled. All labels contain the following information: job number, sample number, client sample ID, , and date/time of sample collection,. Labeling samples is a critical step in the log-in process. Care must be taken to ensure client sample ID's and lab ID's match, sample storage locations are correct, the container count and type is correct. Mislabeling can result in incorrect testing being run on inappropriate samples.

11.3.1.1. Labels that read "Caution-Use Hood!" shall be affixed to all containers for a given sample that are thought to be a safety hazard (for example, high in contaminants, flammable, etc.), or known to emit noxious fumes or odors. The Sample Receiving group is notified of potential hazards by the PM, COC, quote, or client.

11.3.1.1.1. Samples that are known or expected to contain high concentration of Cyanide (250 ppm or more) or Sulfide (500 ppm or more) need to be unpacked in a fume hood. The Sample Receiving group must put a special sticker on these sample bottles indicating to the Lab Groups

that the samples are high in either Cyanide or Sulfide so the Lab Groups can take the necessary safety precautions.

11.4. Storage

11.4.1. Once all sample containers have been properly labeled, the Sample Receiving technician will place the samples into the proper storage locations. These locations are as follows:

- Organic extractable samples are placed in the walk-in refrigerators located in Sample Receiving.
- Volatile samples are stored in the double-door refrigerator located in the Sample Return area.
- Samples known or suspected to be hazardous are stored in a cage in the walk-in cooler and/or in a fume hood in the Volatile rooms.
- Inorganic samples are stored in the walk-in refrigerators located in Sample Receiving.
- Preserved metal samples are placed in a non-refrigerated room. Metals samples that need to be lab filtered and/or preserved are stored in the walk-in cooler. **LLHg samples are stored in the Metals Dept., and MeHg samples are stored in the MeHg cooler in the Sample Return area for the Specialty Analysis Group.**

11.5. Paperwork

11.5.1. All paperwork (COC, CRF, LIMS log-in sheets) associated with a project is clipped together and referred to as a Summary.

11.5.2.

11.6. Bottle Returns

11.6.1. All bottle returns are done by the analyst.

11.6.2.

11.7. Subcontracting of Samples

11.7.1. Samples that are logged for parameters performed at other laboratories are subcontracted to these facilities (including other TestAmerica labs).

11.7.2. A Sample Analysis Requisition (ICOC) is printed for these samples upon completion of the log-in process.

11.7.3. The ICOC contains information necessary for sample analysis. The original form accompanies the samples to the subcontracted laboratory, and a copy is attached to the Summary. The ICOC form must have a relinquished signature with a date and time. Any additional information necessary for sample analysis must be handwritten on the form (e.g. list of compounds, homogenizing of samples, limited quantity, etc.). In order to track subcontracted samples, the lab Purchase Order number on the ICOC form must be recorded in the subcontracted sample PO book located in the Sample Receiving log-in area..

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. Training Qualifications

13.1.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. The Nonconformance Memo shall be filed in the project file.

13.1.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

13.1.3 The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.1.4 The only personnel authorized to execute this SOP are those in the Sample Receiving Dept.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2. PPE required for purging sample wastes include a lab coat, face shield, and gloves.

15.3. All samples may be disposed of 30 days after the report date except for those samples associated with special client retention. Shelves are purged in chronological order. All jobs on a specific shelf must be noted. If a job can be disposed, a disposal date must be recorded.

15.4. Disposal dates are recorded on this printout. When clearing shelves, samples that cannot be disposed of because of special client retention are boxed or stored on carts. Stored samples must have a specific date listed in which samples can be disposed of, or a note that indicates "SAVE" and client name or reason.

15.5. A printout of all lots assigned to certain shelves in each storage location can be obtained from LIMS.

15.6. Solid samples that are non-regulated waste are placed in 55-gallon drum containers for proper disposal.

15.7. Regulated solid waste is placed in the "Hazardous Soils" 55-gallon drum.

15.8. Oils, waste, and solvent samples go into the "Sample Waste" 55-gallon drum.

15.9. Water samples designated for disposal are placed on carts and disposed of in Sample Receiving. Acidified samples are poured into a drum and neutralized under a fume hood in Sample Receiving.

15.10. Solvent waste must be disposed of in clearly labeled waste cans.

16. REFERENCES

16.1 References

16.1.1 TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.2 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.3 Corporate Quality Management Plan (CQMP), current version

16.1.4 Revision History

Historical File:		Revision 6.0: 09/02/99		Revision 6.6: 09/18/09
		Revision 6.1: 06/14/01		Revision 6.7: 12/14/09
		Revision 6.2: 10/06/03		Revision 6.8: 08/18/10
		Revision 6.3: 04/20/04		
		Revision 6.4: 10/02/06		
		Revision 6.5: 07/01/08		

16.2 Associated SOPs and Policies, current version

16.2.1 QA Policy, QA-003

16.2.2 Supplemental Practices for DoD Project Work, NC-QA-016

17. MISCELLANEOUS

17.1 Appendices

17.1.1 Appendix I – Example of Expirables

17.1.2 Appendix II – Example of Cooler Receipt Form

17.1.3 Appendix III – Example of Sample Containers, Preservatives, Holding Times

17.1.4 Appendix IV – Example of Preservatives

Appendix I – Example of Expirables

TESTAMERICA NORTH CANTON

EXPIRABLES

24-HOUR HOLD TIME:

pH – WATER (9040/4500/150.1—must be on bottle)

*Cr^{+6} – WATER (7196/3500—must be on bottle)

FERROUS IRON

RESIDUAL CHLORINE

DISSOLVED OXYGEN (DO)

CORROSIVITY – WATER

48-HOUR HOLD TIME:

BOD/CBOD

*NO_2 – IC (300/9056) or TRACCS (353)

*NO_3 – IC (300/9056) or TRACCS (353)

*OPO_4 – IC (300/9056) or MANUAL (365/4500)

ALKALINITY – TOTAL, CARB, BI-CARB, HYDROXIDE

TURBIDITY

CORROSIVITY – SOLID

SETTLEABLE SOLID

*CR^{+6}, NO_2, NO_3, AND OPO_4 ARE VERY IMPORTANT.
THESE SHOULD BE DONE FIRST!!!

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Appendix II – Example of Cooler Receipt Form

TestAmerica Cooler Receipt Form/Narrative **Lot Number:** _____
North Canton Facility

Client _____ Project _____ By: _____
Cooler Received on _____ Opened on _____ (Signature)
FedEx ☐ UPS ☐ DHL ☐ FAS ☐ Stetson ☐ Client Drop Off ☐ TestAmerica Courier ☐ Other _____
TestAmerica Cooler # _____ Multiple Coolers ☐ Foam Box ☐ Client Cooler ☐ Other _____

1. Were custody seals on the outside of the cooler(s)? Yes ☐ No ☐ Intact? Yes ☐ No ☐ NA ☐
   If YES, Quantity _____
   Were custody seals on the outside of cooler(s) signed and dated? Yes ☐ No ☐ NA ☐
   Were custody seals on the bottle(s)? Yes ☐ No ☐
   If YES, are there any exceptions? _____
2. Shippers' packing slip attached to the cooler(s)? Yes ☐ No ☐
3. Did custody papers accompany the sample(s)? Yes ☐ No ☐ Relinquished by client? Yes ☐ No ☐
4. Were the custody papers signed in the appropriate place? Yes ☐ No ☐
5. Packing material used: Bubble Wrap ☐ Foam ☐ None ☐ Other _____
6. Cooler temperature upon receipt _____ °C See back of form for multiple coolers/temps ☐
   METHOD: IR ☐ Other ☐ _____
   COOLANT: Wet Ice ☐ Blue Ice ☐ Dry Ice ☐ Water ☐ None ☐
7. Did all bottles arrive in good condition (Unbroken)? Yes ☐ No ☐
8. Could all bottle labels be reconciled with the COC? Yes ☐ No ☐
9. Were sample(s) at the correct pH upon receipt? Yes ☐ No ☐ NA ☐
10. Were correct bottle(s) used for the test(s) indicated? Yes ☐ No ☐
11. Were air bubbles >6 mm in any VOA vials? Yes ☐ No ☐ NA ☐
12. Sufficient quantity received to perform indicated analyses? Yes ☐ No ☐
13. Was a trip blank present in the cooler(s)? Yes ☐ No ☐ Were VOAs on the COC? Yes ☐ No ☐

Contacted PM _____ Date _____ by _____ via Verbal ☐ Voice Mail ☐ Other ☐
Concerning _____

14. CHAIN OF CUSTODY

The following discrepancies occurred:

_____
_____
_____
_____
_____
_____

15. SAMPLE CONDITION

Sample(s) _____ were received after the recommended holding time had expired.
Sample(s) _____ were received in a broken container.
Sample(s) _____ were received with bubble >6 mm in diameter. (Notify PM)

16. SAMPLE PRESERVATION

Sample(s) _____ were further preserved in sample receiving to meet recommended pH level(s). *Nitric Acid Lot#* 113007-HNO_3; *Sulfuric Acid Lot#* 071707-H_2SO_4; *Sodium Hydroxide Lot#* 073007 -*NaOH; Hydrochloric Acid Lot#* 092006-*HCl; Sodium Hydroxide and Zinc Acetate Lot#* 050205-CH_3COO_2ZN/*NaOH.*
What time was preservative added to sample(s)? _____

Client ID	pH	Date	Initials

SOP: NC-SC-0005, Sample Receiving
N:\QAQC\NARRATIV\TestAmerica\Cooler Receipt TestAmerica\COOLER_TestAmerica_Rev 66 033108.doc

Appendix III – Example of Sample Containers, Preservatives, Holding Times

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative [1,2]	Volume	Parameter	Container	Preservative [1,2]	Volume
601/602	G	HCl	3x40 mL	PCB	G	None	2 L
Acidity	P	None	250 mL	Pesticides	G	None	2 L
Alkalinity (Sep)	P	None	250 mL	Pesticides + PCBs	G	None	2 L
Amenable Cyanide	P	NaOH	250 mL	pH	P	None	50 mL
Ammonia Nitrogen (NH_3)	P	H_2SO_4	500 mL	Phenols	G	H_2SO_4	1 L
Asbestos	P	None	250 mL	PNA/PAH	G	None	2 L
Bicarbonate	P	None	250 mL	R. Chlorine	P	None	100 mL
BNA + Dioxin	G	None	2 L	Radiological Alpha, Beta, Radium	P	HNO_3	4 L
BNAs	G	None	2 L	Reactive Cyanide	P	None	1 L
BOD	P	None	250 mL	Reactive Sulfide	P	None	1 L
Bromide (Br)	P	None	250 mL	Settleable Solids	P	None	1L
BTEX & MTBE	G	HCl	3x40 mL	Silica	P	None	250 mL
BTEX 8021	G	HCl	3x40 mL	Sulfate	P	None	250 mL
Carbonaceous BOD	P	None	250 mL	Sulfide	P	Zn Acetate & NaOH	1 L
Carbonate	P	None	250 mL	Sulfite	P	None	250 mL
Chemical Oxygen Demand	P	H_2SO_4	250 mL	Surfactants (MBAS)	P	None	250 mL
Chloride (Cl)	P	None	250 mL	T. Coliform	P	None	125 mL
Chromium, $^{6+}$	P	None	250 mL	TDS	P	None	250 mL
COD	P	H_2SO_4	250 mL	TKN	P	H_2SO_4	1L
Color	P	None	50 mL	TON	P	H_2SO_4	1 L
Conductivity	P	None	250 mL	Total Cyanide	P	NaOH[3]	250 mL
Corrosivity	P	None	250 mL	Total Organic Carbon (TOC)	G	HCl	2 x40 mL
Dissolved Metals*	P	HNO_3	1 L	Total Organic Halogens	G	H_2SO_4	250 mL
Dissolved Oxygen	G	None	300 mL	Total Phosphorus	P	H_2SO_4	250 mL
Elemental PO_4	G	None	250 mL	Total Solids	P	None	250 mL
Fecal Coliform	P	None	125 mL	TPH - Diesel (Ext.)	G	None	2 L
Flashpoint	G	None	100 mL	TPH - Gasoline (P&T)	G	HCl	2x40 mL
Fluoride	P	None	250 mL	TPH-GC	G	None	2 L
Formaldehyde	G	None	500 mL	TRPH - IR 418.1	G	HCl	2 L
Free Cyanide	P	NaOH	250 mL	TSS	P	None	250 mL
Glycols 8015	G	None	2x40 mL	Turbidity	P	None	250 mL
Hardness	P	HNO_3	250 mL	TVS	P	None	250 mL

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative [1,2]	Volume	Parameter	Container	Preservative [1,2]	Volume
Herbicides	G	None	2 L	VOC	G	HCl	3x40 mL
Metals	P	HNO_3	1 L	VOC 601	G	HCl	3x40 mL
Nitrate	P	None	250 mL	VOC 602	G	HCl	3x40 mL
Nitrate/Nitrite	P	H_2SO_4	250 mL	VOC 624	G	HCl	8x40 mL
Nitrite	P	None	250 mL	VOC 624	G	HCl	3x40 mL
Oil & Grease	G	H_2SO_4	1 L	VOC 8260	G	HCl	3x40 mL
OPPs	G	None	2 L	VOC and VOA	G	HCl	3x40 mL
Orthophosphate	P	None	250 mL				

Notes:

* Filtered in field

[1] HCl, HNO_3, and H_2SO_4 to pH < 2. NaOH to pH > 12

[2] Temperature = ≤ 6°C except for aqueous metals

Appendix IV – Example of Preservatives

PRESERVATIVES

NITRIC ACID	**< 2**	**HNO_3**	**Add Nitric Acid to Contents After Filling**
SULFURIC	**< 2**	**H_2SO_4**	**Add Sulfuric Acid to Contents After Filling**
SODIUM HYDROXIDE	**> 12**	**NAOH**	**Add Sodium Hydroxide to Contents After Filling**
HYDROCHLORIC ACID	**< 2**	**HCl**	**Add Hydrochloric Acid to Bottle Prior to Filling**
			Add Zinc Acetate to Contents After Filling
ZINC ACETATE/SODIUM	**> 9**	**ZNACE/NAOH**	
			Add Sodium Hydroxide to Contents After Filling

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Appendix G

Field Methods Standard Operating Procedures

Appendix G.1

Surface Water Sampling
Standard Operating Procedures

Table of Contents

Section 1.0 Planning and Preparation

Prior to surface water sampling:

1. Review the Work Plan, project documents, and Site-Specific Health and Safety Plan (HASP) with the Project Manager/Coordinator.
2. Review the Quality Assurance Project Plan (QAPP) with the Project Coordinator and Project Chemist to determine Quality Assurance/Quality Control (QA/QC) and decontamination requirements.
3. Complete a Field Equipment Requisition Form (QSF-014). The Project Planning, Completion, and Follow-Up Checklist (Form SP-02) should be used for guidance throughout the project.
4. Confirm with the Project Manager/Coordinator that a Property Access/Utility Clearance Data Sheet (QSF-019) has been completed.
5. Arrange access to the site. Consider site access conditions (e.g., snow).
6. For surface water sampling consider if hazards exist due to deep/fast moving water, difficult access, and if additional CRA personnel are required for safety and health reasons.
7. Contact the CRA Chemistry group to arrange:
   - SSOW (Simplified Scope of Work)
   - Laboratory
   - Sample containers delivery
   - Preservatives if required
   - Filtration information if required
   - Coolers
   - Shipping details
   - Sample starting date
   - Expected duration of sampling program
8. Evaluate sample notification needs with the Project Coordinator. Have the regulatory groups, client, landowner, CRA personnel, and laboratory been notified of the sampling activities?
9. Plan the sequence of sampling activities to reduce the potential for cross-contamination. For surface water sampling, start downstream and progress upstream.

Section 2.0 Quality Assurance/Quality Control

For Quality Assurance/Quality Control (QA/QC) purposes, CRA will submit one field duplicate, one trip blank, and one MS/MSD for analyses for every 10 surface water samples analyzed.

Section 3.0 Equipment Decontamination

Equipment decontamination between sampling locations will not be required as CRA field personnel will use new sample tubing at each sample location. Upon completion of the sampling program, measurement tools (e.g., yard sticks, survey rods) may be cleaned as follows:

1. Wash with clean potable water and laboratory detergent, using a brush as necessary to remove particulates
2. Rinse with tap water
3. Rinse with deionized water
4. Air dry for as long as possible

Section 4.0 Field Procedures for Surface Water Sampling

4.1 General

Surface water sampling is performed to obtain samples for surface water bodies that are representative of existing surface water conditions.

Surface water sampling locations for surface water quality and groundwater interaction studies are selected based on the following:

1. Study objectives
2. Location of point surface discharges
3. Non-point source discharges and tributaries
4. Presence of structures (e.g., bridge, dam)
5. Accessibility

During surface water sampling it is important to obtain samples that are not impacted by the re-suspension of sediment produced because of improper or poor surface water sampling techniques.

4.2 Surface Water Sample Location Selection

Bridges and piers are good locations for surface water sampling locations since they provide easy access and permit water sampling across the entire width of the surface water body. The JSA for sampling from bridges must include a traffic management plan to assure the employee has considered using a spotter, signage, cones, and flags to warn car traffic of the work adjacent to the roadway. Wading for surface water samples increases the chances of disturbance of sediments from the floor of the surface water body.

When wading for surface water samples in lakes, ponds, streams, and slow moving rivers be aware of potential safety and health risks. A life vest and safety line must be worn at all times where footing is unstable or when sampling in fast moving water or water that is more than 3 feet (0.9 m) deep. A two-person team is required for most surface water sampling activities, a Project Manager must approve a one person sampling team. If the site conditions require the use of a life vest and safety line, the two people involved in the sampling must be competent swimmers.

Surface water samples must be collected with no suspended sediments. Surface water samples are collected commencing with the furthest downstream location to avoid sediment interference with upstream locations and are collected facing upstream.

Section 5.0 Sampling Equipment and Techniques

As per Section 3.3 of the FSP, CRA will collect surface water samples at each area using pre- cleaned sample containers (US-DH-81 sampler of equivalent) by filling the containers at a low flow rate (<100 mL/min) using a peristaltic pump and new disposable tubing. Prior to sampling, CRA will determine the mid-point of the water column using an appropriate measurement tool (i.e., yard stick, survey rod, or equivalent). Surface water samples will be collected from approximately the mid-point of the water column. Sample collection will require the sampling tubing to be tethered to a survey pole at the appropriate water column depth. For sampling in the GMR, if possible, the sampling personnel will stay at the water's edge so as not to disturb the water during sample collection. If sampling from the edge of the water is not possible, personnel will wade into the GMR or enter the GMR on a watercraft to sample, depending on the water depth. Wading or movement may cause sediment deposits to be re-suspended and can result in biased samples and is acceptable if the stream has a noticeable

current and the samples are collected directly in the sample container when faced upstream. Surface water sampling in the Quarry Pond will require the use of watercraft. All sample volumes collected for analyses of VOCs and dissolved metals will be collected using a peristaltic pump (as described above) to minimize aeration while allowing for sample preservation.

All surface water samples will be analyzed for hardness, and field parameters (pH, temperature, Oxidation Reduction Potential (ORP), and conductivity) at the sampling locations using a YSI Model 3560 instrument. Additionally, Dissolved Oxygen (DO) will be measured using a YSI Model 52 instrument, and turbidity will be measured using an HF Scientific DRT-15C Turbidimeter. Alternatively, equivalent instruments may be used.

Section 6.0 Field Notes for Surface Water Sampling

Use a standard CRA field book to record daily surface sampling activities, describe surface water sampling locations, sampling techniques, and, if applicable, provide a description of photographs taken. Visual observations are important and provide valuable information when interpreting surface water quality results. Observations include:

1. Weather conditions
2. Stream flow directions
3. Stream physical conditions (width, depth, etc.)
4. Tributaries
5. Effluent discharges
6. Impoundments
7. Bridges
8. Railway trestles
9. Oil sheens
10. Odors
11. Buried debris
12. Vegetation
13. Algae
14. Fish and other aquatic life
15. Surrounding industrial areas

The following factors should be considered for surface water sampling:

1. Predominant Surrounding Land Use: Observe the prevalent land use type in the vicinity and note any other land uses in the area which, although not dominant, may potentially affect surface water quality.
2. Local Watershed Erosion: Note the existing or potential erosion of soil in the local watershed and its movement into the stream. Erosion can be rated through visual observation of watershed stream characteristics including increases or decreases in turbidity.
3. Local Watershed Non-Point Source Pollution: This refers to problems or potential problems other than erosion and sedimentation. Nonpoint source pollution can be diffuse agricultural and urban runoff. Other factors may include feed lots, wetlands, septic systems, dams, impoundments, and mine seepage.
4. Estimated Stream Width: The estimated distance from shore at a transect representative of the stream width in the area.
5. Estimated Stream Depth: Riffle (rocky area), run (steady flow area), and pool (still area). Estimate the vertical distance from the water surface to the bottom of the surface water body at a representative depth at three locations.
6. High Water Mark: Estimate the vertical distance from the bank of the surface water body to the peak overflow level, as indicated by debris hanging in bank or flood plain vegetation, and deposition of silt. In instances where bank flow is rare, high water marks may not be evident.
7. Velocity: Record or measure the stream velocity in a representative run area, if required.
8. Dam Present: Indicate the presence or absence of a dam upstream or downstream of the surface water sampling location. If a dam is present, include specific information detailing the alteration of the surface water flow.
9. Channelized: Indicate if the area surrounding the surface water sampling location is channelized.
10. Canopy Cover: Note the general proportion of open to shaded areas which best describes the amount of cover at the surface water sampling location.

Section 7.0 Follow-Up Activities

The following should be performed once surface water sampling is completed:

1. Double check the Work Plan and QAPP to ensure all samples and QA/QC samples have been collected and confirm with the Project Coordinator.
2. Decontaminate all equipment at the site then return clean to the appropriate office equipment manager.
3. Dispose of decontamination fluid as specified in the Work Plan.
4. Notify the contract laboratory when the samples should arrive. Enclose a completed chain-of-custody in each cooler.
5. Complete and file the appropriate forms and data sheets. Also file the field notes.
6. Return site keys.

Section 8.0 References

For additional information pertaining to surface water sampling, the user of this manual may reference the following:

ASTM D5358 Practice for Sampling with a Dipper or Pond Sampler

ASTM D4489 Practices for Sampling of Waterborne Oils

ASTM D3325 Practice for the Preservation of Waterborne Oil Samples

ASTM D4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents

ASTM D4411 Guide for Sampling Fluvial Sediment in Motion

ASTM D4823 Guide for Core-Sampling Submerged, Unconsolidated Sediments

ASTM D3213 Practice for Handling, Storing, and Preparing Soft Undisturbed Marine Soil

ASTM D3976 Practice for Preparation of Sediment Samples for Chemical Analysis

ASTM E1391 Guide for Collection, Storage, Characterization, and Manipulation of Sediments for Toxicological Testing

ASTM D4581 Guide for Measurement of Morphologic Characteristics of Surface Water Bodies

ASTM D5906 Guide for Measuring Horizontal Positioning During Measurements of Surface Water Depths

ASTM D5073 Practice for Depth Measurement of Surface Water

ASTM D5413 Test Methods for Measurement of Water Levels in Open-Water Bodies

Appendix G.2

Sediment Sampling Standard Operating Procedures

Table of Contents

Section 1.0 Introduction

Sediment sampling is conducted to obtain samples that are representative of existing chemical and/or physical conditions of sediment and sludge. Through assessment of existing site conditions, the distribution of the constituents of concern can be determined and the potential for exposure and risks can be evaluated, including natural attenuation rates and the potential for ongoing sources. The information developed during this evaluation can then be used to determine the most appropriate type of remedial approach for the site, including no action.

Section 2.0 Background

Sampling locations for sediment studies may be selected based on many factors, including: study objectives; tidal conditions; flow conditions; the location of point source discharges, non-point source discharges, and tributaries; the presence of structures (bridges, dams, etc.); and accessibility.

Sediment and sludge samples are collected to determine the nature of discharge impacts on sediment quality. Contaminants may become sediment bound through either deposition of contaminated suspended sediments or by adsorption of soluble surface water constituents to sediments. Many organic chemicals are generally adsorbed to a greater degree as the fraction of organic carbon (foc) in the sediment increases.

It should be noted that sediment and sludge quality can vary substantially with depth depending on flow rates and depositional history. It is critical that sediment and sludge samples be collected from the sampling horizon identified in the Work Plan. If there is any ambiguity regarding the sediment sampling rationale, all concerns must be discussed with the Project Coordinator.

Note: It is important to obtain samples that are unimpacted by the re-suspension of sediment caused by sampling activities.

Section 3.0 Prior Planning and Preparation

The following shall be considered prior to sediment sampling.

1. Review the Work Plan and Site-Specific Health and Safety Plan (HASP), project documents, historic site reports, and a site plan to become familiar with the site.

2. Surrounding area. Review and become familiar with the health and safety requirements, and discuss the work activities with the Project Coordinator.
3. Complete an Equipment Requisition Form (QSF-014) and assemble all required equipment, materials, log books, and forms. The Equipment and Supply Checklist - Sediment Sampling (Form SP-17) provides a summary of the equipment and materials typically required for Sediment Sampling. Form SP-02, Project Planning Completion, and Follow-Up can be used for guidance throughout the project.
4. Review the sampling locations. Sampling locations may, at times, be located on private lands. Coordination of property access should always be discussed with the Project Coordinator in advance of field activities. Have all parties been notified that sampling is scheduled? Has a Property Access/Utility Clearance Data Sheet (QSF-019) been initiated/completed for this field activity?
5. Complete a Vendor Evaluation Form (QSF-012) and file in the Project file for any Vendors that do not have full approval status or are not listed on Approved Vendor List (QSL-004). Completion of a Safety and Health Schedule (QSF-030 for Canadian Work; QSF-031 for U.S. Work) is necessary for all Vendors who complete field services. Prior to mobilization on site, the Vendor must submit the form to the Regional Safety and Health Manager for review and approval (if not already posted on QSL-004).
6. Contact the CRA Chemistry Group to arrange:
   - SSOW (Simplified Scope of Work)
   - laboratory
   - sample containers
   - coolers
   - shipping details
   - sampling start date
   - expected sampling duration
7. Consider in advance of sampling whether a hazard exists due to deep/fast moving water, difficult access, and if more than two people are required. A two-person team is required for most surface water sampling activities; a Project Manager must approve a one-person sampling team. If the site conditions require the use of the life vest and safety line, the two people involved in the sampling must be competent swimmers.
8. Pre-plan the sampling sequence such that sampling commences at the furthest downstream sampling location and proceeds upstream.
9. If sampling within a navigable waterway, determine navigational requirements, and coordinate with the regulatory body (e.g., harbor master, U.S. Army Corps of Engineers).

Note: If sampling on a boat, consider space requirements for Exclusion Zone (EZ), Contaminant Reduction Zone (CRZ), and Support Zone (SZ). Also consider requirements including restrooms, weather protection, communication, etc.

Section 4.0 Quality Assurance/Quality Control

CRA will submit one field duplicate, one trip blank, and one MS/MSD for analyses for every 10 soil samples analyzed. Additionally, CRA will submit 1 equipment blank for every 10 decontamination activities, or at least once per day of sampling equipment cleanings, whichever is more frequent.

Section 5.0 Equipment Decontamination

On environmental sites, sediment sampling equipment (e.g., split spoons, trowel, spoons, shovels, bowls, dredges, corers, scoops) are typically cleaned as follows:

1. Wash with clean potable water and laboratory detergent, using a brush as necessary to remove particulates.
2. Rinse with tap water.
3. Rinse with deionized water.
4. Air dry for as long as possible.

Additional or different decontamination procedures may be necessary if sampling for some parameters, including volatile organic compounds (VOCs) and metals.

Section 6.0 Sample Site Selection

Before any sampling is conducted, the first requirement is to consider suitable sampling locations. Sampling locations should be selected in accordance with the Work Plan and discussed with the Project Coordinator. Wading for sediment samples in lagoons, lakes, ponds, slow-moving rivers, and streams must be done with caution since bottom deposits are easily disturbed. Sampling must only be attempted where safe conditions exist and samples must be collected from undisturbed sediments. All sediment samples are to be collected commencing with the most downstream sample to avoid sediment interference with other downstream samples. A life vest and safety line should be worn in all cases where footing is unstable or where water is fast moving or over 3 feet (0.85 m) in depth. A second person may also be required for most of the sampling scenarios.

6.1 Rivers, Streams, and Creeks

Sediment samples may be collected along a cross-section of a river or stream in order to adequately characterize the bed material, or from specific sediment deposits as described in the Work Plan. A common procedure is to sample at quarter points along the cross-section of the sampling site selected. Samples may be composited as described in the Work Plan.

Samples of dissimilar composition (e.g., grain size, organic content) should not be combined.

Representative samples can usually be collected in portions of the surface water body that have a uniform cross-section and flow rate. Since mixing is influenced by turbulence and water velocity, the selection of a site immediately downstream of a riffle area (e.g., fast flow zone) are likely areas for deposition of sediment since the greatest deposition occurs where stream velocity slows.

6.2 Lakes, Ponds, and Impoundments

The number of sampling sites on a lake, pond, or impoundment will vary with the purpose of the investigation, as well as the size and shape of the basin. Sample selection should adequately represent the conditions of the basin. Attention must be given to identify intakes and outflows within the basin that may provide biased sample representation. Sample locations with adjacent structures (i.e., banks, piers, etc.) may also provide biased samples within active lagoons or settling ponds.

When collecting sediment samples in lakes, ponds, and reservoirs, samples should be collected at approximately the center of the water body or as directed by the Work Plan. This is also the case for reservoirs that are formed by the impoundment of rivers or streams. The coarse-grained sediments are deposited near the headwaters of the reservoir, and the fine-grained sediments near the center. The shape, inflow pattern, and circulation must be considered when selecting sediment sampling sites in lakes and reservoirs.

In all instances, the sampling locations should be properly documented with field notes and photographs, as appropriate.

Section 7.0 Sediment Sampling Equipment and Techniques

CRA will collect two sediment samples from each location. One sample will be collected from the upper (available) layer of sediments (0 - 6 inches below sediment/water interface), and a second sample will be collected from subsurface sediment (greater than 6 inches below

sediment/water interface). The sampling units are individual grab samples. Prior to collection of samples within the GMR, CRA will survey the shore along the Site, and according to the proposed samples locations will wade into the near shore with a probe, to measure water level, and to probe the sediments to locate areas with fine sediments. Sample locations will be adjusted such that samples will be biased towards locations with fine-grained sediments with higher organic carbon. Within the GMR, two sediment samples will also be collected (as above) in depositional locations immediately downstream of any point discharges identified between the upstream dam and the southern Site boundary. Prior to collection of samples within the quarry pond, CRA will visually survey the perimeter of the Quarry Pond. If any evidence of potential leachate migration is observed, two sediment samples will also be collected (as above) in depositional areas of the potential migration locations.

As surface water and sediment samples are being collected at the same locations, surface water samples will be collected first, at each location. When required, a flat bottom boat will be used to access the sediment sampling locations and complete the sampling. Two types of sediment sampling equipment will be available for sample collection. The preferred method of sample collection is a Piston Corer, which is ideal for collecting samples of soft, unconsolidated sediments. The Piston Corer is constructed of cellulose acetate butryrate (CAB) tubing, a piston/seal assembly, and a deployment pole/handle and rope. CAB tubing is an impact resistant plastic that is ideal for scientific studies. The Piston Corer can collect core samples in 2-foot or 4-foot lengths. If significant debris, vegetation, or other material causes refusal using the Piston Corer, the sample location will be off-set up to three times. If the Piston Corer is unsuccessful after three attempts, a stainless steel hand auger corer will be utilized. The stainless steel hand auger corer can collect cores with lengths up to 18 inches. Three CRA staff will be on Site at all times that personnel are on the water. Following retrieval of the sample device, the core will be removed and examined.

Acceptance criteria for sediment core samples are as follows:

- The core penetrated to target depth (12 inches)
- The core did not suffer significant sample-induced compaction or loss of material (i.e., recovery greater than 60 percent, as measured by recovery length divided by penetration length)
- Cored material did not extend out the top of the core tube or contact any part of the sampling apparatus at the top of the core tube
- There are no obstructions in the cored material that might have blocked the subsequent entry of sediment into the core tube, which may have resulted in an incomplete and biased core section

Once the cores have been collected from all the proposed locations, the cores will be processed on land. The following steps outline the general procedures to be followed when cores are split, logged, and subsampled for laboratory analysis:

1. All equipment coming into contact with sediment will be decontaminated before use with each sample to avoid cross contamination.
2. Cut the core liner longitudinally on opposite sides using a small jig or reciprocating saw. Pull away the top half of the core liner to expose the sediment sample.
3. Photograph the core, including a sign identifying the core location.
4. Log and describe the sediment on a core log form according to standard ASTM soil description procedures. Core logs should include:
    a. Visual grain size classification
    b. Color
    c. Consistency (stiffness or denseness)
    d. Odor
    e. Presence of debris
5. After the sediment description is complete, subsample the core in 0-6 inch and 6-12 inch intervals (based on in situ conditions).
6. After the subsampling is complete, fill sample jars for VOC analysis.
7. Homogenize the remaining soil from each depth interval using a stainless steel mixing spoon or an electric drill with a stainless steel paddle.
8. Collect samples of the homogenized sediment as appropriate for chemical analysis. Label sample jars and place them in refrigerators or coolers with blue ice to maintain samples at 4°C until dispatched under chain of custody to the appropriate laboratory.

7.2 Air Monitoring

Prior to sediment/sludge sampling, measure the breathing space above the sample location with a photoionization detector (PID), should the potential for volatiles be present, and use a hydrogen sulfide meter should hydrogen sulfide be present. Repeat these measurements during sampling. If either of these measurements exceed any of the air quality criteria established in the HASP, air purifying respirators (APRs) or supplied air systems will be required.

Hydrogen sulfide odors are typical in lagoons and settling ponds where decomposition with depth occurs over time.

7.3 Sample Location Tie-In/Surveying

The recording of the sample locations and depth on the site plan is extremely important. This may be accomplished by manual measurement (i.e., swing ties), global positioning system (GPS) survey, or stadia methods. Manual measurements for each sample location should be tied into three permanent features (e.g., buildings, utility poles, hydrants). Diagrams with measurements should be included in the field book.

> *Note: Manual field measurements are always necessary regardless of whether a survey is completed. Manual measurements allow future identification of the sample location without the need of a survey crew to locate positions using a grid system.*

7.4 Field Notes

A bound field book is used to record daily activities, describe sampling locations and techniques, and describe photographs (if taken). Visual observations are important, as they may prove invaluable in interpreting water or sediment quality results. Observations shall include (as applicable) weather, stream flow conditions, stream physical conditions (width, depth, etc.), tributaries, effluent discharges, impoundments, bridges, railroad trestles, oil sheens, odors, buried debris, vegetation, algae, fish or other aquatic life, and surrounding industrial areas. The following observations should be considered:

- **Predominant Surrounding Land Use**: Observe the prevalent land use type in the vicinity (noting any other land uses in the area which, although not predominant, may potentially affect water quality).
- **Local Watershed Erosion**: The existing or potential erosion of soil within the local watershed (the portion of the watershed that drains directly into the stream) and its movement into a stream is noted. Erosion can be rated through visual observation of watershed and stream characteristics. (Note any turbidity observed during water quality assessment.)
- **Local Watershed Non-point Source Pollution**: This item refers to problems and potential problems other than siltation. Non-point source pollution is defined as diffuse agricultural and urban runoff (e.g., stormwater runoff). Other compromising factors in a watershed that may affect water quality are feedlots, wetlands, septic systems, dams and impoundments, and/or mine seepage.
- **Estimated Stream Width**: Estimate the distance from shore at a transect representative of the stream width in the area.

- **Estimated Stream Depth**: Riffle (rocky area), run (steady flow area), and pool (still area). Estimate the vertical distance from water surface to stream bottom at a representative depth at each of the three locations.
- **High Water Mark**: Estimate the vertical distance from the stream bank to the peak overflow level, as indicated by debris hanging in bank or floodplain vegetation, and deposition of silt or soil. In instances where bank overflow is rare, a high water mark may not be evident.
- **Velocity**: Record an estimate of stream velocity in a representative run area (see Section 12.0).
- **Dam Present**: Indicate the presence or absence of a dam upstream or downstream of the sampling station. If a dam is present, include specific information relating to alteration of flow.
- **Channelized**: Indicate whether the area around the sampling station is channelized.
- **Canopy Cover**: Note the general proportion of open to shaded area which best describes the amount of cover at the sampling station.
- **Sediment Odors**: Disturb sediment and note any odors described (or include any other odors not listed) which are associated with sediment in the area of the sampling station.
- **Sediment Oils**: Note the term which best describes the relative amount of any sediment oils observed in the sampling area.
- **Sediment Characteristics:** Note the grain size, color, consistency, layering, presence of biological organisms, man-made debris, etc. in accordance with standard ASTM soil description protocols.
- **Sediment Deposits**: Note those deposits described (or include any other deposits not listed) which are present in the sampling area. Also indicate whether the undersides of rocks not deeply embedded are black (which generally indicates low dissolved oxygen or anaerobic conditions).

Section 8.0 Follow-Up Activities

The following shall be performed once field activities are complete.

1. Equipment shall be cleaned and returned to the equipment administrator and the appropriate form dated and signed.
2. The contracted laboratory shall be notified as to when to expect sample arrival. The sample cooler shall contain the chain-of-custody form.
3. Field notes shall be sent to file and the field book stored at the CRA office.

4. At the completion of the sediment sampling program, the Project Planning, Completion and Follow-Up Checklist (Form SP-02) must be completed to document activities conducted and serves to remind personnel of the various tasks required. This form must be signed and filed at the respective field office, regional CRA office and issued to the Project Coordinator.

Section 9.0 References

For additional information pertaining to this topic, the user of this manual may reference the following:

ASTM D5358 Practice for Sampling with a Dipper or Pond

Sampler ASTM D4489 Practices for Sampling of Waterborne Oils

ASTM D3325 Practice for the Preservation of Waterborne Oil Samples

ASTM D4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents

ASTM D4416 Guide for Sampling Fluvial Sediment in Motion

ASTM D4823 Guide for Core-Sampling Submerged, Unconsolidated Sediments

ASTM D3213 Practice for Handling, Storing, and Preparing Soft Undisturbed Marine

Soil ASTM D3976 Practice for Preparation of Sediment Samples for Chemical Analysis

ASTM E1391 Guide for Collection, Storage, Characterization, and Manipulation of Sediments for Toxicological Testing

ASTM D4581 Guide for Measurement of Morphologic Characteristics of Surface Water Bodies

ASTM D5906 Guide for Measuring Horizontal Positioning During Measurements of Surface Water Depths

ASTM D5073 Practice for Depth Measurement of Surface Water

ASTM D5413 Test Methods for Measurement of Water Levels in Open-Water Bodies